# SIEMENS WATER TECHNOLOGIES CORP. CARBON REACTIVATION FURNACE RF-2 PERFORMANCE DEMONSTRATION TEST DATA VALIDATION REPORT

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#### 1.0 INTRODUCTION

On March 28 through 30, 2006, samples were collected during a Performance Demonstration Test (PDT) of the Siemens water technologies Corp. Carbon Regeneration Unit (RF-2). The samples were analyzed by Sevren Trent Laboratories, Knoxville, TN, Severn Trent Laboratories, Sacramento, CA, MVA Scientific Consultants, Duluth, GA, Galbraith Laboratories, Inc., Knoxville, TN, and Airtech Environmental Services, Inc., Arvada, CO. The types of samples analyzed are noted below.

#### Airtech

```
Stack Gas Particulates - Gravimetric (EPA Method 5)
Stack Gas Total Volatile Organics (Bag) – GS/FID (SW846 0040 Guidance for Total Organics)
```

# Severn Trent Laboratories

```
Process Total Chlorine – IC ( SW846-9056 )
Stack Gas Volatiles – GC/MS ( SW846 5041 & 8260 )
Stack Gas HCI/Cl2 – IC ( EPA 26A )
Process Volatiles – GC/MS ( SW846 8260 )
Process Semivolatile Organics – GC/MS ( SW846 8270 )
Process Metals – ICP/CVAA ( SW846 6010/7470 & 7471 )
Stack Gas Metals – ICPMS/CVAA ( SW846 6010/7470 )
```

Stack Gas Dioxin/Furans - HRGC/HRMS ( SW846 0023A/ 8290 )

Stack Gas Total Volatile Organics (Con) GC/FID( SW846 0040 Guidance for Total Organics )

Stack Gas Semivolatile Organics – GC/MS ( SW846 8270 ) Stack Gas Organochlorine Pesticides – GC ( SW846 8081 )

Stack Gas Polychlorinated Biphenyls – HRGC/HRMS (EPA 1668)

Stack Gas Polynuclear Aromatic Hydrocarbons – HRGC/LRMS (STL SOP 0016)

Stack Gas Total Semivolatile & Nonvolatile Organics - GC/FID & Grav ( EPA 18 Guidance for Total Organics )

Stack Gas Hexavalent Chromium - IC (SW846 0061/7199)

# MVA

Particle Size distribution (Scanning Electron Microscopy)

# Galbraith

Process Ultimate Analysis (ASTM Method D5373)

The results of these analyses were received by Focus Environmental, Inc. and have been reviewed for data quality. Specific quality control guidelines are discussed preceding the data evaluation in each section. These guidelines include, however are not limited to, the following areas:

# **Contract Compliance Screening (CCS)**

CCS provides an assessment of the laboratory's conformance to deliverable and other contract/quality assurance provisions.

# **Compliance with Sample Handling Criteria**

Failure to meet holding time or other sample handling and preservation criteria can result in a low bias to the data and possible false negatives.

# **Instrument Performance Criteria**

These criteria include GC/MS tuning requirements, internal standard retention time and retention time shift criteria, general chromatographic performance, and other broad indications of instrument performance.

# **Initial and Continuing Calibrations**

Failure to meet calibration response, retention time, linearity, and stability criteria can result in quantitative biases, false positives and negatives, and misidentification of analytes.

# **Precision Measurements**

Precision measurements include field and laboratory duplicates (example: a MS/MSD) as well as comparisons of spike recoveries (surrogates) across multiple samples. Failure to meet precision criteria can indicate that the quantitative results may be variable and not be representative of actual field conditions.

# **Accuracy Measurements**

Accuracy measurements include recovery of various spikes (examples: MS and LCS), calibration verifications, and performance demonstrations (example: Audit samples). Failure to meet accuracy criteria may indicate that quantitative results are biased or are indicative of interferences in the analysis.

## **Blanks**

Blanks demonstrate that the analytical result is an actual representation of field conditions and not the result of cross contamination of the sample from the environment, laboratory, or other sources.

#### **Qualitative and Quantitative Results**

On an audit basis, the identification and the quantitation of results reported by the laboratory are verified to ensure appropriateness and completeness.

#### 2.0 STACK GAS DIOXIN/FURAN

Stack gas dioxin/furan data were received as one package. Samples G-2937/2938-R1-FH, G-2939/2940-R1-BH, G-3049/3050-R2-FH, G-3051/3052-R2-BH, G-3128/3129-R3-FH, G-3130/3131-R3-BH, G-3132/3133-R3-FH-BT, G-3134/3135-R3-BH-BT, and G-3136-XAD-RB were included. These samples were prepared and analyzed using SW846 Method 0023A and SW846 Method 8290. Section 2.1 provides a list of the primary data quality objectives evaluated during this review. Section 2.2 summarizes the significant findings of the evaluation.

#### 2.1 DATA QUALITY OBJECTIVES

# 2.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings</u>: All sample reporting was provided as needed to confirm analytical results.

## 2.1.2 Sample Handling Criteria

<u>Requirements</u>: Samples must be chilled. Samples are to be held no more than 30 days from sampling to extraction and no more than 45 days from extraction to analysis.

<u>Findings</u>: All samples were chilled as required, extracted within 30 days, and analyzed within 45 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

#### 2.1.3 Instrument Performance Criteria

<u>Requirement:</u> Retention time window verification and GC column performance should be checked at the beginning of each 12-hour shift and should meet the requirements of Section 8.2.1 of SW-846 Method 8290.

<u>Findings</u>: Retention time windows and GC column performance were checked at the beginning of each 12-hour shift.

# 2.1.4 Initial and Continuing Calibrations

Requirements: Five high-resolution concentration calibration solutions should be used for the initial calibration.

The initial calibration relative standard deviations (RSDs) for the mean relative response factor (RRF) from the 17 unlabeled standards should not exceed  $\pm$  20%, and those for the nine labeled reference compounds must not exceed  $\pm$  30%.

A midlevel standard should be run at the beginning of each 12-hour shift. The continuing calibration response factors (RFs) should be within  $\pm$  20% of the initial calibration mean RRF for unlabeled standards and  $\pm$  30% for the labeled standards.

<u>Findings</u>: Five standards were used for the initial calibration. All labeled and unlabeled calibration standards met the required RSD requirements.

Six continuing calibration midlevel standards were analyzed in association with these samples. All labeled and unlabeled calibration standards met the required percent difference (%Ds) requirements.

# 2.1.5 Precision Objectives

- < 30% RSD of spike recoveries between samples for labeled compounds spiked prior to sampling.
- < 60% RSD of spike recoveries between samples for internal quantitation standards.

<u>Findings:</u> All of the front half and back half internal standard and surrogate recovery RSDs were within the required limits.

# 2.1.6 Accuracy Objectives

Requirements: 70 – 130% recovery of isotopically labeled PCDD/PCDF compounds spiked onto each sorbent resin tube prior to sampling

- 40 130% recovery of isotopically labeled tetra through hexa chlorinated PCDD/PCDF internal quantitation standards spiked onto train components prior to extraction.
- 25 130% recovery of isotopically labeled hepta and octa chlorinated PCDD/PCDF internal quantitation standards spiked onto train components prior to extraction

<u>Findings:</u> The 13C-1,2,3,4,6,7,8-HpCDD internal standard recovery for the Run 3 front half sample reported a slightly high recovery. All other surrogate and internal standard recoveries were reported within the established limits. The blank train front half sample also reported one surrogate recovery exceeding the criteria.

#### 2.1.7 Blanks

<u>Requirements</u>: Once during each test, a blank train is set up in the field and recovered in the same manner as other field samples. Analysis of the blank train is performed to assess contamination.

Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be conducted to assess contamination

<u>Findings:</u> A blank train was set up in the field and samples collected and analyzed with the other field samples. Also, method blanks were analyzed as required with the samples. Additionally media checks were analyzed in association with these samples. Sample results were compared to determine if any positive sample results were reported at levels less than five times the highest associated blank result. Below is a list of the positive results reported for the blanks and positive sample results reported less than five times the highest associated blank result.

#### **Positive Blank Results**

G-3132/3133-R3-FH-BT G-3134/3135-R3-BH-BT OCDD 20 BJ OCDF 2.4 QBJ

G-3136-R3-XAD-RB Total HxCDD OCDD	1.4 J 6.5 BJ	A-5379-Media Check XAD OCDD OCDF	13 BJ 2.5 QBJ
A-5381-Media Check F OCDD OCDF	ilter 16 BJ 2.8 QBJ		
Method Blank (79) (FH	/Filter)	Method Blank (92) (BH/XAD)	
1,2,3,7,8,9-HxCDD	0.80 QJ	1,2,3,4,6,7,8-HpCDD	2.1 J
Total HxCDD	0.80 QJ	Total HpCDD	2.1 J
OCDD	17 J	OCDD	15 J
2,3,4,6,7,8-HxCDF	1.0 QJ	2,3,4,6,7,8-HxCDF	1.2 J
Total HxCDF	1.0 QJ	1,2,3,7,8,9-HxCDF	1.1 J
1,2,3,4,6,7,8-HpCDF	1.2 QJ	Total HxCDF	2.3 J
1,2,3,4,7,8,9-HpCDF	1.1 J	1,2,3,4,6,7,8-HpCDF	1.3 QJ
Total HpCDF	2.3 QJ	Total HpCDF	1.3 QJ
OCDF	4.5 J	OCDF	5.3 J

# Field Sample Results less than Fve Times Associated Blanks

Run 1 Front Half	Run 1 Back Half
------------------	-----------------

OCDD OCDD Total HpCDF OCDF

OCDF

Run 2 Front Half Run 2 Back Half

Total HpCDF OCDF

Run 3 Front Half Run 3 Back Half

1,2,3,4,6,7,8-HpCDF OCDD Total HpCDF OCDF

**OCDF** 

# 2.1.8 Qualitative and Quantitative Results

Requirement: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors or any additional issues noted during this review.

Results were qualified by the laboratory using a "Q" or "J" qualifier. The "J" qualifier indicates that the sample result is greater than the estimated detection limit (EDL) and less than the reporting limit (minimum level). Results with concentrations should be considered estimated. The "Q" qualifier indicates that not all of the chromatographic data requirements were met as described in the case narrative for this data package. Results qualified with a "Q" by the laboratory should be considered estimated.

#### 2.2 SIGNIFICANT FINDINGS

There were 522 quality control criteria evaluated for this analysis. 494 of the evaluated criteria were found to meet the project objectives and 28 were found to be outside the control limits.

The 13C-1,2,3,4,6,7,8-HpCDD internal standard recovery for the Run 3 front half sample reported a slightly high recovery. The total HpCDD and positive isomer results should be considered estimated and possibly biased high.

There were positive results reported for both the blank train samples and the method blanks. The sample results were compared to the blank results and the sample results reported as less than five times the blank result were listed in section 2.1.7. These results should be considered estimated, biased high.

#### 3.0 STACK GAS VOLATILE ORGANICS

The volatile stack gas data were received in one package. Samples G-2910-R1-P2-T, G-2911-R1-P2-T/C, G-2912-R1-P3-T, G-29131-R1-P3-T/C, G-2914-R1-P4-T, G-2915-R1-P4-T/C, G-2916-R1-CON, G-2917-R1-T-FB, G-2918-R1-T/C-FB, G-3008-R2-P2-T, G-3009-R2-P2-T/C, G-3010-R2-P3-T, G-3011-R2-P3-T/C, G-3012-R2-P4-T, G-3013-R2-P4-T/C, G-3014-R2-CON. G-3015-R2-T-FB, G-3016-R2-T/C-FB, G-3089-R3-P1-T, G-3090-R3-P1-T/C, G-3091-R3-P2-T, G-3092-R3-P2-T/C, G-3093-R3-P3-T, G-3094-R3-P3-T/C, G-3095-R3-P4-T, G-3096-R3-P4-T/C, G-3097-R3-CON, G-3098-R3-T-FB, G-3099-R3-T/C-FB, G-3100-R3-T-TB, G-3101-R3-T/C-TB, and G-3102-R3-DI Water-TB, were included. Tube samples were prepared using SW846 Method 5041A and both VOST tubes and condensate samples were analyzed according to SW846 Method 8260B. Section 3.1 provides a list of the primary data quality objectives evaluated during this review. Section 3.2 summarizes the significant findings of the evaluation.

# 3.1 DATA QUALITY OBJECTIVES

# 3.1.1 Contract Compliance Monitoring

<u>Requirement:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

# 3.1.2 Sample Handling Criteria

Requirements: Samples must be chilled. Samples are to be held no more than 14 days.

<u>Findings</u>: All samples were chilled as required and analyzed within 14 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, stack-sampling personnel did not sign the chain of custody documentation.

## 3.1.3 Instrument Performance Criteria

<u>Requirements</u>: Internal standards spiked into each sample, standard and, blank should report a retention time within 30 seconds and an area within – 50 to + 100% of the last calibration check.

<u>Findings:</u> All three internal standards were within the specified quality control criteria for retention time and area for all field samples.

# 3.1.4 Initial and Continuing Calibrations

Requirements: Three to five standards bracketing the expected concentration should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF for the CCCs should not exceed + 30%.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each 12-hour shift. The continuing calibration RFs should be within  $\pm$  30% of the initial calibration mean RRF for CCCs.

<u>Findings</u>: The original initial calibration associated with the VOST tubes and the extended calibration curve for chlorobenzene and tetrachloroethene for the VOST tubes reported all SPCCs, CCCs, and POCHs within the required quality control criteria. All other target compounds for these initial calibrations also reported %RSDs less than 30%. The initial calibration associated with the condensate samples reported all SPCCs, CCCs, and POHCs with the required quality control criteria. All other target compounds for these initial calibrations also reported %RSDs less than 30%.

There were three continuing calibrations associated with these samples. All of the SPCCs, CCCs, and POHCs reported %Ds within the specified quality criteria. There were a few target compounds in each continuing calibration that reported %Ds greater than 25%. Although this was not a specified quality criterion, positive results associated with these compounds should be considered estimated. Below is a list of the compounds founds with elevated %Ds. Positive tube results associated with these elevated %Ds were the bromomethane results for G-2911-R1-P2-T/C, G-2913-R1-P3-T/C, G-2915-R1-P4-T/C, G-2918-R1-T/C-FB, G-3009-R2-P2-T/C,G-3011-R2-P3-T/C, G-3013-R2-P4-T/C, G-G-3016-R2-T/C-FB, G-3092-R2-P2-T/C, G-3094-R3-P3-T/C, G-3096-R3-P4-T/C, G-3098-R3-T-FB, G-3099-R3-T/C-FB, G-3100-R3-T-TB, and G-3101-R3-T/C-TB. The only positive condensate results associated with these elevated %Ds were the acetone results for G-2916-R1-CON, G-3014-R2-CON, G-3097-R3-CON, and G-3102-R3-DI Water TB.

# **Elevated Continuing Calibration Percent Differences**

Continuing Calibration 4/5/06 Bromomethane Carbon Tetrachloride 1,1,1,2-Tetrachloroetha 1,2-Dibromo-3-chloropr	ne	26.2% 29.3% 25.1% 34.5%	Tubes
Continuing Calibration 4/7/06 Bromomethane	07:35	28.0%	Tubes
Continuing Calibration 4/7/06 Acetone 2,2-Dichloropropane 4-Methyl-2-Pentanone 1,3,5-Trimethylbenzene n-Butylbenzene Naphthalene	09:16 97.5% 34.7% 25.1% 30.3% 36.1% 48.8%		Condensate

# 3.1.5 Precision Objectives

Requirements: < 25% RPD between the spike recoveries from four VOST tubes analyzed prior to sample analysis.

< 35% RSD of spike recoveries between each field sample.

<u>Findings:</u> The RSDs between the field samples for each surrogate were within the quality criteria for both the VOST tubes and the condensate samples. The RPDs for 2-butanone (30%) and 1,2-Dibromo-3-chloropropane (27%) for the tenax/charcoal tube pair were reported slightly above the quality criteria. There were no positive results associated with these compounds therefore there is no action required.

# 3.1.6 Accuracy Objectives

Requirements: 75 – 125% recovery of standards spiked onto two tube sets prior to sample analysis

50 – 150% recovery of isotopically labeled surrogates spiked onto every field sample.

EPA Audit sample results should be within 50 – 150% of the true value.

<u>Findings</u>: All field sample surrogate recoveries were within the quality criteria except sample G-3095-R3-P4-T .G-3089-R3-P1-T. Sample G-3095-R3-P4-t reported the bromofluorobenzene surrogate recovery at 49%. The fourth set of tubes was analyzed for this run. Sample G-3089-R3-P1-T also reported the bromofluorobenzene surrogate recovery at 49%. The three other surrogates for these two samples reported recoveries within the specified criteria and sample results are equivalent to the other two tube pairs. No action is required.

The LCS associated with the tube samples (spiked VOST tubes) reported several compounds outside the 75 – 125% recovery requirement. All positive results associated with these recoveries should be qualified as estimated and either biased high or low depending on the recovery reported. Below is a list of the spiked tube recoveries reported outside the quality criteria and a list of the positive field sample results associated with these recoveries. All of the listed results except chloromethane, bromomethane, and iodomethane associated with the tenax/charcoal results are biased low. The tenax/charcoal results for bromomethane, chloromethane, and iodomethane are biased high.

## Spiked Tube Recoveries Outside The Quality Criteria

Tenax Spiked Tube Pair		Tenax/Charcoal Tube Pair	
Acetone	128 / 127	Acetone	50 / 63
Acrylonitrile	53 / 54	Acrylonitrile	71
Bromobenzene	70 / 74	Bromomethane	139 / 129
Bromoform	56 / 59	2-Butanone	60
2-Butanone	57 / 67	Chloroethane	128 / 129
Chlorodibromomethane	64 / 69	Chloromethane	171 / 162
1,2-Dibromo-3-chloropropane	30 / 34	1,2-Dibromo-3-chloropropane	50 / 67
1,2-Dibromoethane	59 / 62	Iodomethane	129
1,2-dichlorobenzene	62 / 64	Naphthalene	62
1,3-Dichlorobenzene	72 / 72	Trichlorofluoromethane	130
1,4-dichlorobenzene	70 / 72		
1,3-dichloropropane	66 / 69		
2,2-Dichloropropane	62 / 65		
trans-1,3-Dichloropropene	62 / 68		
Hexachlorobutadiene	74		
2-Hexanone	57 / 67		
4-Methyl-2-pentanone	68		
Styrene	73		
1,1,1,2-Tetrachloroethane	63 / 66		
1,1,2,2-Tetrachloroethane	57 / 60		
1,2,3-Trichlorobenzene	73		
1,2,4-Trichlorobenzene	69		
1,1,2-Trichloroethane	64 / 67		
1,2,3-Trichloropropane	51 / 54		

Associated Positive Field Sample Results

G-2910-R1-P2-T G-2911-R1-P2-T/C Bromoform Acetone

Chlorodibromomethane Bromomethane

1,1,1,2-Tetrachloroethane

Chloroethane Chloromethane

Bromomethane

Chloromethane Iodomethane

G-2912-R1-P3-T

Acetone **Bromoform** 

Chlorodibromomethane

G-29131-R1-P3-T/C

G-2914-R1-P4-T

Acetone **Bromoform**  G-2915-R1-P4-T/C

Acetone

Acetone

**Bromomethane** Chloromethane Iodomethane

Trichlorofluoromethane

G-3008-R2-P2-T

Acetone **Bromoform** 

Chlorodibromomethane

G-3009-R2-P2-T/C

Acetone

Bromomethane Chloromethane Iodomethane

G-3010-R2-P3-T

Acetone **Bromoform** 

Chlorodibromomethane

G-3011-R2-P3-T/C

Acetone

**Bromomethane** Chloromethane Iodomethane

G-3012-R2-P4-T

Acetone **Bromoform** 

Chlorodibromomethane

G-3013-R2-P4-T/C

Acetone

Bromomethane Chloromethane Iodomethane

G-3089-R3-P1-T

Acetone

**Bromoform** 

Chlorodibromomethane

G-3090-R3-P1-T/C Chloromethane

G-3091-R3-P2-T

Acetone **Bromoform** 

Chlorodibromomethane

G-3092-R3-P2-T/C

Acetone

Bromomethane Chloromethane Iodomethane

G-3093-R3-P3-T

Acetone **Bromoform** 

Chlorodibromomethane

G-3094-R3-P3-T/C

Acetone

**Bromomethane** Chloromethane Iodomethane

G-3095-R3-P4-T

Acetone **Bromoform** 

Chlorodibromomethane

G-3096-R3-P4-T/C

Acetone

**Bromomethane** Chloromethane Iodomethane

#### **3.1.7** Blanks

<u>Requirements</u>: One set of field blank tubes should be analyzed for every test run. The results should be less than the lowest calibration standard.

One set of trip blank tubes should accompany each tube shipment from the field and should be analyzed if the field blank shows contamination. The results should be less than the lowest calibration standard.

One set of laboratory blank tubes (method blanks) should be analyzed with each analytical batch. The results should be less than the lowest calibration standard.

System blanks are analyzed daily before sample analysis and between high level samples. The results should be less than the lowest standard.

<u>Findings</u>: There were a few positive results reported in the field blanks, trip blanks, media checks and method blanks. These results were compared to the field sample results and the positive results less than five times the associated highest blank should be considered estimated biased high. Below are lists of the positive results found in the various blanks associated with these samples and of samples with results less than five times the highest concentration found in the associated blanks

#### Positive Blank Results

Run 1 FB Tenaxk Acetone Iodomethane	0.099 JB4 0.013 JB	Run 1 FB Tenax / Chard Acetone Bromomethane Chloromethane Iodomethane	0.10 B 0.025 JB 0.0053 J 0.014 JB
Run 2 FB Tenax Acetone Bromomethane Carbon Disulfide Iodomethane	0.089 JB 0.024 JB 0.0026 0.014 JB	Run 2 FB Tenax / Chara Acetone Bromomethane Chloromethane Iodomethane	coal 0.096 JB 0.030 JB 0.18) 0.016 JB)
Run 3 FB Tenax Acetone Bromomethane Iodomethane	0.094 JB 0.025 JB 0.014 JB	Run 3 FB Tenax / Charcoal Acetone 0.094 JB Bromomethane 0.027 JB Chloromethane 0.016 J Iodomethane 0.014 JB	
TB Tenax Acetone Bromomethane Iodomethane	0.092 JB 0.023 JB 0.013 JB	TB Tenax / Charcoal Acetone Bromomethane Chloromethane Iodomethane	0.039 JB 0.026 JB 0.029 0.015 JB
Media Check Tenax Acetone Bromomethane Iodomethane	0.094 JB 0.025 JB 0.014 JB	Media check Tenax / Cl Acetone Bromomethane Chloromethane Iodomethane	narcoal 0.094 JB 0.029 JB 0.011 0.014 JB
Media check A-5388 (6) Acetone	096033) (T/C) 0.098 JB	Method Blank (6096033 Acetone	3) (T/C) 0.095 J

Benzene 0.0078 J Bromomethane 0.026 J Bromomethane 0.026 JB Iodomethane 0.014 J

lodomethane 0.014 JB

Method Blank (6097054) (tenax) 1,2,4-Trichlorobenzene 0.0030 J

DI Water Trip Blank
Acetone
4.1 J
Bromodichloromethane
2.7
Bromoform
0.86 J
Method Blank (6095253) (condensate)
lodomethane
0.55 J
1,2,3-Trichlorobenzene
0.26 J
1,2,4-Trichlorobenzene
0.15 J

Chlorodibromomethane 1.1
Chloroform 8.0
Methylene chloride 1.2 J
Trichloroethene 0.91 J

Results Less Than Five Times the Associated Blank Result

G-2910-R1-P2-T G-2912-R1-P3-T G-2914-R1-P4-T

Acetone Acetone Acetone

G-2911-R1-P2-T/C G-29131-R1-P3-T/C G-2915-R1-P4-T/C

Acetone Acetone Bromomethane Bromomethane Bromomethane Iodomethane Iodomethane

Iodomethane

G-2916-R1-CON-A G-2916-R1-CON-B

Acetone Acetone

Bromodichloromethane

Chloroform

Bromoform Chlorodibromomethane

Methylene chloride
Trichloroethene

G-3008-R2-P2-T G-3010-R2-P3-T G-3012-R2-P4-T

Carbon Disulfide Acetone Acetone

G-3009-R2-P2-T/C G-3011-R2-P3-T/C G-3013-R2-P4-T/C

AcetoneAcetoneAcetoneBromomethaneBromomethaneBromomethaneChloromethaneChloromethaneChloromethaneIodomethaneIodomethaneIodomethane

G-3014-R2-CON-A G-3014-R2-CON-B

Acetone Acetone Methylene chloride Iodomethane

G-3089-R3-P1-T G-3091-R3-P2-T

Acetone Acetone

G-3090-R3-P1-T/C G-3092-R3-P2-T/C

Benzene Acetone Bromomethane

Chloromethane lodomethane

G-3093-R3-P3-T G-3095-R3-P4-T

Acetone Acetone

G-3094-R3-P3-T/C G-3096-R3-P4-T/C

Acetone Acetone

Bromomethane Bromomethane Chloromethane Iodomethane Iodomethane

G-3097-R3-CON-A G-3097-R3-CON-B,
Acetone Bromodichloromethane

Acetone

Methylene chloride Bromoform

lodomethane Chlorodibromomethane

Chloroform

Methylene chloride Trichloroethene

#### 3.1.8 Qualitative and Quantitative Results

<u>Requirements:</u> Separate analysis of front and back tubes from each set should show less than 30% of the front tube concentration on the back tube. This criterion is not applicable for a particular compound if the back tube contains less than 75 ng of the compound.

The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> The results for the tenax/charcoal tubes were compared to the corresponding tenax tubes to determine if there was any breakthrough. None of the tenax/charcoal tubes reported results 30% or more of the tenax tube concentration when the tenax tube reported 75 ng or more of the compound.

Compounds were reported with a "J" qualifier by the laboratory. This qualifier indicates that the sample concentration was greater than the method detection limit and less than the reporting limit. Results "J" qualified by the laboratory should be considered estimated.

During the analysis of the VOST samples G-2910-R1-P2-T, G-2912-R1-P3-T, G-3090-R3-P1-T, G-3091-R3-P2-T, and G-3093-R3-P3-T reported results exceeding the calibration range. Since these samples could not be reanalyzed at a dilution of the original analysis, the laboratory set up an extended calibration curve for the affected compounds and requantified the samples as discussed in the case narrative of the VOST analytical data package. The extended curve showed good linearity and the requantified continuing calibration reported good percent differences for the compounds. The laboratory also spiked blank tenax tubes at levels higher than any of the reported samples. The recoveries for these spiked tubes were within the required 75 - 125% recovery for tetrachloroethene (80 / 80%) and below the criteria for chlorobenzene (61 / 63%). The requantified results were all less than the original results, therefore the original results should be qualified as estimated biased high. The original and requantified results for all results reported exceeding the calibration range are listed below.

Original and Requantified Results

G-2910-R1-P2-T

Chlorobenzene Tetrachloroethene	5.8 E 4.7 E	5.2 4.2
G-2912-R1-P3-T Chlorobenzene	3.5 E	3.2
G-3089-R3-P1-T Chlorobenzene	2.3 E	2.1
G-3091-R3-P2-T Chlorobenzene Tetrachloroethene	3.4 E 2.4 E	3.1 2.2
G-3093-R3-P3-T Chlorobenzene	3.1 E	2.8

# 3.2 SIGNIFICANT FINDINGS

There were 2572 quality control criteria evaluated for this analysis. 2448 of the evaluated criteria were found to meet the project objectives and 124 were found to be outside the control limits.

There were three continuing calibrations associated with these samples. All of the SPCCs, CCCs, and POHCs reported %Ds within the specified quality criteria. There were a few target compounds in each continuing calibration that reported %Ds greater than 25%. Although this was not a specified quality criterion, positive results associated with these compounds should be considered estimated. Below is a list of the compounds founds with elevated %Ds. Positive tube results associated with these elevated %Ds were the bromomethane results for G-2911-R1-P2-T/C, G-2913-R1-P3-T/C, G-2915-R1-P4-T/C, G-2918-R1-T/C-FB, G-3009-R2-P2-T/C, G-3011-R2-P3-T/C, G-3013-R2-P4-T/C, G-G-3016-R2-T/C-FB, G-3092-R2-P2-T/C, G-3094-R3-P3-T/C, G-3096-R3-P4-T/C, G-3098-R3-T-FB, G-3099-R3-T/C-FB, G-3100-R3-T-TB, and G-3101-R3-T/C-TB. The only positive condensate results associated with these elevated %Ds were the acetone results for G-2916-R1-CON, G-3014-R2-CON, G-3097-R3-CON, and G-3102-R3-DI Water TB.

All field sample surrogate recoveries were within the quality criteria. The LCS associated with the tube samples (spiked VOST tubes) reported several compounds outside the required 75 – 125% recovery requirement. All positive results associated with these recoveries should be qualified as estimated and either biased high or low depending on the recovery reported. A list of the spiked tube recoveries reported outside the quality criteria and a list of the positive field sample results associated with these recoveries are included in section 3.1.6. All of the listed results except chloromethane, bromomethane, and iodomethane associated with the tenax/charcoal results are biased low. The tenax/charcoal results for bromomethane, chloromethane, and iodomethane are biased high.

There were a few positive results reported in the field blanks, trip blanks, media checks and method blanks. These results were compared to the field sample results and the positive results less than five times the associated highest blank should be considered estimated biased high. A list of the positive results found in the various blanks associated with these samples and a list of samples with results less than five times the highest concentration found in the associated blanks are included in section 3.1.7

During the analysis of the VOST samples G-2910-R1-P2-T, G-2912-R1-P3-T, G-3090-R3-P1-T, G-3091-R3-P2-T, and G-3093-R3-P3-T reported results exceeding the calibration range. Since these samples could not be reanalyzed at a dilution of the original analysis, the laboratory set up an extended calibration curve for the affected compounds and requantified the samples as discussed in the case narrative of the VOST analytical data package. The extended curve showed good linearity and the requantified continuing calibration reported good percent differences for the compounds. The laboratory also spiked blank tenax tubes at levels higher than any of the reported samples. The recoveries for these spiked

tubes were within the required 75-125% recovery for tetrachloroethene (80 / 80%) and below the criteria for chlorobenzene (61 / 63%). The requantified results were all less than the original results, therefore the original results should be qualified as estimated biased high. The original and requantified results for all results reported exceeding the calibration range are listed in section 3.1.8.

#### 4.0 STACK GAS HYDROGEN CHLORIDE AND CHLORINE

The stack gas hydrogen chloride and chlorine data were received as one package. Samples G-2987-R1-H2SO4, G-2979-R1-NAOH, G-2982-R1-H2SO4-RB, G-2983-R1-NAOH-RB, G-3065-R2-H2SO4, G-3066-R2-NAOH, G-3149-R3-H2SO4, and G-3150-R3-NAOH were included. The samples were prepared and analyzed for hydrogen chloride or chlorine following EPA Method 26A and SW846 Method 9057 as appropriate for the matrix by ion chromatography. Section 4.1 provides a list of the primary data quality objectives evaluated during this review. Section 4.2 summarizes the significant findings of the evaluation.

#### 4.1 DATA QUALITY OBJECTIVES

# 4.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings</u>: All sample reporting was provided as needed to confirm analytical results.

# 4.1.2 Sample Handling Criteria

Requirements: Samples are to be held no more than 30 days.

<u>Findings:</u> All samples were analyzed within 30 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, stack-sampling personnel did not sign the chain of custody documentation.

# 4.1.3 Initial and Continuing Calibrations

<u>Requirements:</u> Initial calibration - Four standards bracketing the expected concentration should be used for the initial calibration. Linear correlation coefficient > 0.995.

Continuing calibration – Analyzed at the beginning and end of the analysis period and after every ten samples. Should be within 90 - 110 % of the theoretical value.

<u>Findings</u>: The initial calibration was performed using 6 standards and the linear correlation coefficient was greater than 0.995. Continuing calibration verifications were analyzed at the rate required and all reported recoveries within the criteria.

# 4.1.4 Precision Objectives

Requiments: The RPD of duplicate analysis should be < 25%.

<u>Findings</u>: All RPDs were within the required criterion.

# 4.1.5 Accuracy Objectives

Requirement: 85 – 115% recovery of chloride spiked into an aliquot of both acidified and alkaline impinger solution.

<u>Findings</u>: The matrix spike / matrix spike duplicate (MS/MSD) using acidified impinger solution reported recoveries within the required limits. However, both of the MS/MSDs using the alkaline impinger solutions reported recoveries exceeding the specified limits. These matrix spiked were analyzed at successive dilutions with the recoveries improving with the dilution until the point where the sample was over diluted. The recoveries were never within limits. All eight laboratory control samples reported recoveries within the specified criteria indicating the analysis is in control. The high recoveries for the alkaline impingers indicate a matrix affect. The alkaline impinger results should be considered estimated biased high.

# 4.1.6 Blanks

<u>Requirements:</u> Analysis of one set of reagent blanks and a method blank carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.

<u>Findings:</u> Reagent blanks and method blanks were analyzed as required with all results reported as non detect.

#### 4.1.7 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

# 4.2 SIGNIFICANT FINDINGS

There were 43 quality control criteria evaluated for this analysis. 39 of the criteria were found to meet the project objectives and 4 were found to be outside control limits.

The matrix spike / matrix spike duplicate (MS/MSD) using acidified impinger solution reported recoveries within the required limits. However, both of the MS/MSDs using the alkaline impinger solutions reported recoveries exceeding the specified limits. These matrix spiked were analyzed at successive dilutions with the recoveries improving with the dilution until the point where the sample was over diluted. The recoveries were never within limits. All eight of the laboratory control samples reported recoveries within the specified criteria indicating the analysis is in control. The high recoveries for the alkaline impingers indicate a matrix affect. The alkaline impinger results should be considered estimated biased high.

#### 5.0 STACK GAS PARTICULATE

The stack gas particulate data were received as one data package. Samples Run 1-Filter, Run 1-Rinse, Run 2-Filter, Run 2-Rinse, Run 3-Filter, and Run 3-Rinse were included. The samples were analyzed gravimetrically following the requirements of EPA Methods 5 and 26A. Applicable compliance areas were reviewed and with no significant findings. Section 5.1 provides a list of the primary data quality objectives evaluated during this review. Section 5.2 summarizes the significant findings of the evaluation.

#### 5.1 DATA QUALITY OBJECTIVES

# 5.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

#### 5.1.2 Calibration Check

Requirements: The balance should be checked prior to analysis with standardized weights and should be within 99 -101 % agreements

<u>Findings:</u> The balance was checked prior to analysis and all weights were within 99 – 101% agreement.

# 5.1.3 Precision Objectives

Requirements: Duplicate weighing of each sample should be within 0.5 mg or 1% total tare weight whichever is greater.

<u>Findings</u>: Duplicate weighings of each sample were within 0.5 mg or 1% of the total tare weight.

# 5.1.4 Accuracy Objectives

Requirements: The balance should be checked prior to analysis with standardized weights and should be within 99 -101 % agreements

<u>Findings:</u> The balance was checked prior to analysis and all weights were within 99 – 101% agreement.

# **5.1.5** Blanks

<u>Requirements:</u> Analysis of reagent blanks and method blanks carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.

<u>Findings:</u> Both the filter and the acetone rinse blanks reported nondetect results.

# 5.1.6 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

# 5.2 SIGNIFICANT FINDINGS

There were 11 quality control criteria evaluated for this analysis. 11 of the criteria were found to meet the project objectives and 0 were found to be outside control limits.

#### 6.0 STACK GAS METALS

The stack metals data were received as a single package. Samples G-2953/2954-R1-FH, G-2955-r1-Bh, G-2956-R1-Empty Imp, G-2957-R1-KMNO4 Imp, G-2958-R1-HCI Imp, G-3057/3058-R2-FH, G-3059-R2-BH, G-3060-R2-Empty Imp, G-3061-R2-KMNO4 Imp, G-3062-R2-HCI Imp, G-3141/3142-R3-FH, G-3143-R3-BH, G-3144-R3-Empty Imp, G-3145-R3-KMNO4 Imp, and G-3146-R3-HCI Imp were included. The mercury samples container four separate back half analyses. The samples were prepared by EPA Method 29 and analyzed for total metals by and inductively coupled plasma –mass spectrometry (ICPMS) by SW846 Method 6020 and by for mercury by cold vapor atomic absorption by SW846 Method 7470A. Applicable compliance areas were reviewed and the significant findings discussed below. Section 6.1 provides a list of the primary data quality objectives evaluated during this review. Section 6.2 summarizes the significant findings of the evaluation.

# 6.1.1 Data Quality Objectives

# 6.1.2 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings</u>: Not every mercury sample was analyzed in duplicate. However, the Run 1 impinger samples included matrix spike / matrix spike duplicate analysis in addition to the two spiked blank trains being analyzed in duplicate. This should provide sufficient information to determine precision for this analysis.

# 6.1.3 Sample Handling Criteria

Requirements: Mercury samples are to be held no more than 28 days and ICP samples no more than 180 days.

<u>Findings</u>: All mercury samples were analyzed within 28 days and the ICP samples within 180 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, stack-sampling personnel did not sign the chain of custody documentation.

# 6.1.4 Initial and Continuing Calibrations

<u>Requirements:</u> CVAA – A minimum of five standards bracketing the expected concentration. Correlation coefficient of linear plot > 0.995

ICP and CVAA – Continuing calibrations analyzed at the beginning and end of each analysis period and after every ten samples. Results should be  $\pm$  10% of the theoretical value for the ICP analysis and  $\pm$  20% of the theoretical value for GFAA analysis.

<u>Findings:</u> Both initial calibrations were performed using five standards and a blank and reported correlation coefficients greater than 0.995

# 6.1.5 Precision Objectives

Requirements: Relative percent difference (RPD) between two spiked blank trains should be < 35%

For mercury only, the relative percent difference between the duplicate analysis for one sample should be < 25%

<u>Findings:</u> All ICP laboratory control / laboratory control sample duplicates reported good precision All of the ICP relative percent differences were within limits for the spiked blank train / spiked blank train duplicate except the back half manganese RPD. The spiked blank train duplicate for this analyte reported high recoveries. All positive back half manganese results should be qualified as estimated.

All mercury laboratory control / laboratory control sample duplicates reported good precision. The mercury spiked blank train / spiked blank train duplicate  $KMnO_4$  and HCl impingers reported high RPDs (163 and 114) because the blank spiked train duplicate reported high recoveries (958% and 385%). Contamination is expected in this sample but the source could not be determined. MS/MSDs were analyzed for all three impingers using the Run 1 samples with good precision reported for all results. Results for the  $KMnO_4$  and HCl impingers samples should be qualified as estimated biased high due to possible contamination.

# 6.1.6 Accuracy Objectives

Requirements: Recovery of each metal of concern spiked onto two complete sampling trains should be 70-130%.

Recovery of 70 – 130% for each metal of concern spiked onto an aliquot of each train component.

<u>Findings</u>: All ICP laboratory control samples (LCS) reported acceptable recoveries. All blank spiked train recoveries were also within limits except the blank spiked duplicate result for manganese. This manganese recovery was high (220%). All positive manganese results should be qualified estimated biased high.

All mercury LCSs reported acceptable recoveries. The blank spike trains duplicate  $KMnO_4$  and HCl impingers reported high recoveries (958% and 385%). Contamination is expected in this sample but the source could not be determined. MS/MSDs were analyzed for all three impingers using the Run 1 samples with good recoveries reported for all results. Results for the  $KMnO_4$  and HCl impingers samples should be qualified as estimated biased high due to possible contamination.

Post digestion spikes were analyzed for all front and back half samples. All ICP and mercury post digestion spikes were within limits except the G-3141/3142-R3-FH aluminum recovery. This recovery was reported slightly high (116.4%). This aluminum result should be considered estimated biased high.

# 6.1.7 Blanks

<u>Requirements:</u> Analysis of one set of reagent blanks and a method blank carried through all preparation and analysis steps, used to evaluate contamination.

<u>Findings</u>: All of the appropriate blank samples were analyzed. The front half blank train and the filter media check reported a few positive results. The field sample concentrations were compared to the blank results. All positive results less than five times the highest associated blank concentration should be qualified as estimated biased high. Below are lists of the positive blank results and of the field sample result reported less than five times the blank concentration.

Positive Blank Results

G-2959/2960-R1-FH-BT Aluminum 74.0 ug A-5383-Media Check (Filter) Aluminum 71.2 ug

Antimony	2.7 B ug	Antimony	3.0 B ug
Barium	1.8 B ug	Barium	1.9 B ug
Chromium	0.40 B u	Manganese	0.37 B ug
Copper	1.2 B ug	Nickel	3.6 B ug
Manganese	0.69 B ug	Selenium	1.9 ug
Nickel	3.7 B ug	Thallium	9.8 ug
Selenium	2.0 ug		
Thallium	9.1 ug		

Field Sample Results Less Than Five Times the Blank Concentration

G-2953/2954-R1-FH	G-3057/3058-R2-FH	G-3141-3142-R3-FH
Aluminum	Aluminum	Aluminum
Antimony	Antimony	Antimony
Barium	Barium	Barium
Nickel	Nickel	Nickel
Selenium	Selenium	Selenium
Thallium	Thallium	Thallium

#### 6.1.8 Qualitative and Quantitative Results

4.9 ug

Zinc

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

Compounds were reported with a "B" qualifier by the laboratory. This qualifier indicates that the sample concentration was greater than the method detection limit (MDL) and less than the reporting limit (RL). Results "B" qualified by the laboratory should be considered estimated. It should also be noted that the "J" qualifier typically used to indicate results between the MDL and RL is used to indicate method blank contamination in metals analyses. Blank contamination is addressed in section 6.1.7.

## 6.2 SIGNIFICANT FINDINGS

There were 440 quality control criteria evaluated for this analysis. 418 of the criteria were found to meet the project objectives and 22 were found to be outside control limits.

All ICP laboratory control / laboratory control sample duplicates reported good accuracy and precision All of the ICP relative percent differences and percent recoveries were within limits for the spiked blank train / spiked blank train duplicate except the back half manganese blank train spike duplicate (220%) and RPD (66). All positive back half manganese results should be qualified as estimated.

All mercury laboratory control / laboratory control sample duplicates reported good accuracy and precision. The mercury spiked blank train / spiked blank train duplicate  $KMnO_4$  and HCl impingers reported high RPDs (163 and 114) because the blank spike train duplicate reported high recoveries (958% and 385%). Contamination is expected in this sample but the source could not be determined. MS/MSDs were analyzed for all three impingers using the Run 1 samples with good precision reported for all results. Results for the  $KMnO_4$  and HCl impingers samples should be qualified as estimated biased high due to possible contamination.

All of the appropriate blank samples were analyzed. The front half blank train and the filter media check reported a few positive results. The field sample concentrations were compared to the blank results. All positive results less than five times the highest associated blank concentration should be qualified as estimated biased high. Lists of the positive blank results and of the field sample result reported less than five times the blank concentration are located in section 6.1.7.

Post digestion spikes were analyzed for all front and back half samples. All ICP and mercury post digestion spikes were within limits except the G-3141/3142-R3-FH aluminum recovery. This recovery was reported slightly high (116.4%). This aluminum result should be considered estimated biased high.

#### 7.0 STACK GAS TOTAL VOLATILE ORGANICS

The stack gas total volatile data were received in two packages. The tedlar bag analyses were received in one package and the condensates in another package. Results for Run 1A, Run 1B, Run 2A, Run 2B, Run 3A and, Run 3B bags were included in the tedlar bag results and samples G-2941-R1, G-3053-R2, G-3137-R3, and G-2943-R1-TB were included as the condensates. Samples were analyzed using GC/FID. All applicable compliance areas were reviewed and the significant findings discussed below. Section 7.1 provides a list of the primary data quality objectives evaluated during this review. Section 7.2 summarizes the significant findings of the evaluation.

# 7.1 DATA QUALITY OBJECTIVES

# 7.1.1 Contract Compliance Monitoring

<u>Requirements</u>: Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings</u>: All sample reporting was provided as needed to confirm analytical results. It should be noted that the laboratory adds a surrogate to the condensate samples that is not required by the method but provides additional quality control that would not otherwise be available. The tetraglyme solvent used by the laboratory in preparing these samples is also removed from the results by blank correcting in the C4 results using the peak in the solvent blank.

# 7.1.2 Sample Handling Criteria

Requirements: Condensate samples must be chilled. Samples are to be held no more than 14 days.

Tedlar bags must be analyzed within 72 hours.

<u>Findings</u>: All condesate samples were analyzed within 14 days and the bag samples within 72 hours. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, stack-sampling personnel did not sign the chain of custody documentation.

# 7.1.3 Initial and Continuing Calibrations

<u>Requirements:</u> Minimum three standards bracketing the expected concentration should be used for the initial calibration.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each day. Results should be + 10% of the true value.

<u>Findings:</u> Three standards were used to calibrate for the bag samples and six standards were used to calibrate for the condensate samples. Midlevel standards were analyzed for both analyses with all results within  $\pm$  10%.

#### 7.1.4 Precision Objectives

Requirements: < 35% RPD of concentration between duplicate analysis of a known gas.

<u>Findings</u>: Bag samples and standards were all analyzed in either duplicate or triplicate. All RPDs and RSDs met the required criteria. An LCS/LCSD was analyzed in association with the condensate samples. All RPDs were within the control limits.

# 7.1.5 Accuracy Objectives

Requirements: 80 – 120% recovery of calibration standards spiked into a bag and analyzed prior to sample analysis.

<u>Findings</u>: A standard was spiked into a bag and analyzed each day prior to and following the analyses. All recoveries met the control criteria. An LCS/LCSD was analyzed in association with the condensate samples. All recoveries met the control criteria.

# **7.1.6** Blanks

<u>Requirements:</u> Analysis of one method blank per sample batch of condensate samples carried through all preparation and analysis steps should be less than 20% of sample levels or below the detection limit.

One bag should be filled with zero air or zero nitrogen as a field blank for the bag samples. Results should be less than 20% of the sample level or below the detection limit.

Analyze one aqueous field blank per test condition to assess contamination.

<u>Findings:</u> The required blank were analyzed with the bag and condensate samples. There were no positive results reported in the blanks associated with the bag samples. Positive results for C5 (0.065 JB ug/L) and C6 (2.5 ug/L) were reported in the trip blank. Also, the method blank reported a positive result for C5 (0.31J ug/L). Sample results were compared to the blank results with all positive results for C5 and C6 for the field samples being less than five times the highest blanks. All positive results for C5 and C6 should be qualified estimated biased high.

## 7.1.7 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

Compounds were reported with a "J" qualifier by the laboratory. This qualifier indicates that the sample concentration was greater than the method detection limit and less than the reporting limit. Results "J" qualified by the laboratory should be considered estimated.

## 7.2 SIGNIFICANT FINDINGS

There were 45 quality control criteria evaluated for this analysis. 42 of the criteria were found to meet the project objectives and 5 were found to be outside control limits.

The required blank were analyzed with the bag and condensate samples. There were no positive results reported in the blanks associated with the bag samples. Positive results for C5 (0.065 JB ug/L) and C6

(2.5 ug/L) were reported in the trip blank. Also, the method blank reported a positive result for C5 (0.31J ug/L). Sample results were compared to the blank results with all positive results for C5 and C6 for the field samples being less than five times the highest blanks. All positive results for C5 and C6 should be qualified estimated biased high.

The laboratory adds a surrogate to the condensate samples that is not required by the method but provides additional quality control that would not otherwise be available. The tetraglyme solvent used by the laboratory in preparing these samples is also removed from the results by blank correcting in the C4 results using the peak in the solvent blank.

#### 8.0 STACK GAS SEMIVOLATILES

The stack gas semivolatiles data were received in a single package. Samples G-2919/2930-R1-FH, G-2921/2922-R1-BH, G-2923/2924-R1-CON, G-3017/3018-R2-FH, G-2019/3020-R2-BH, G-3021/3022-R2-CON, G-3023/3024-R2-FH-BT, G-3025/3026-R2-BH-BT, G-3027/3028-R2-CON-BT, G-3029-R2-XAD-RB, G-3103/3104-R3-FH, G-3105/3106-R3-BH, and G-3107/3108-R3-CON were included. These samples were analyzed using SW846 Method 8270. All applicable compliance areas were reviewed and the significant findings discussed below. Section 8.1 provides a list of the primary data quality objectives evaluated during this review. Section 8.2 summarizes the significant findings of the evaluation.

#### 8.1 DATA QUALITY OBJECTIVES

# 8.1.1 Contract Compliance Monitoring

<u>Requirements</u>: Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings</u>: All sample reporting was provided as needed to confirm analytical results.

## 8.1.2 Sample Handling Criteria

<u>Requirements</u>: Samples must be chilled. Samples are to be held no more than 14 days from sampling to extraction and no more than 40 days from extraction to analysis.

<u>Findings</u>: All samples were chilled as required, extracted within 14 days, and analyzed within 40 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

# 8.1.3 Instrument Performance Criteria

Requirements: Internal standard spiked into each sample, standard and, blank should report a retention time within 30 seconds and an area within – 50 to + 100% of the last calibration check.

<u>Findings:</u> The internal standards met the quality control criteria for retention time and area for all field samples.

# 8.1.4 Initial and Continuing Calibrations

Requirements: Five standards bracketing the expected concentration should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF for the CCCs should not exceed ± 30%.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each 12-hour shift. The continuing calibration response factors should be within  $\pm$  30% of the initial calibration mean RRF for CCCs.

<u>Findings:</u> Initial and continuing calibrations were analyzed as required. All relative standard deviations, relative response factors and percent differences were within the required quality control criteria.

# 8.1.5 Precision Objectives

<u>Requirements</u>: <40% RPD (or 35% RSD, if greater than 4 determinations are made) of spike recoveries between field samples.

<50% RPD for duplicate injections from one run. This criterion is relaxed to 100% RPD if the compound is found at a concentration below the lowest standard.

<u>Findings:</u> The 40% RPD of spike recoveries between surrogate requirement was not met by the front half samples because the Run 1 sample reported low recoveries for the surrogates. The RPDs between the Run 2 and Run 3 front half samples when compared without this sample were within the quality criteria. The Run 1 sample only reported one positive result, bis(2-ethylhexyl)phthalate. This sample result should be considered estimated. The condensate surrogates also did not meet this requirement. Positive results were reported for bis(2-ethylhexyl)phthalate in the condensate field samples. These results should be qualified estimated.

There were no duplicate injections of field samples performed. There was however blank spikes analyzed in duplicate of each matrix. The front and back half spiked reported RPDs within the quality control criteria. The condensate blank spike reported the 2,4-dimethylphenol RPD (64) exceeding the quality control criteria. There were no positive results for this compound, therefore no qualification is required.

# 8.1.6 Accuracy Objectives

Requirements: Recovery of isotopically labeled POHCs or appropriate surrogates spiked into each sample as listed in Table 5-2 of the QAPP. If this criterion cannot be met, then the recoveries should be within 3 standard deviations of the historical means of surrogate recoveries for the method (laboratory specific).

Recovery of POHCs or appropriate surrogates spiked onto a blank XAD-2 resin trap in the laboratory as listed in Table 5-2 of the QAPP.

<u>Findings</u>: All front half and back half samples reported surrogate recoveries within the quality control criteria except G-2919/2920-R1-FH. Sample G-2919/2920-R1-FH reported four of the six surrogates below the control limit. Results for this sample should be considered estimated biased low. All of the condensate samples reported at least one base/neutral surrogate exceeding the quality control limit. Samples G-2923/2924-R1-CON, and G-3107/3108-R3-CON reported two bas/neutral surrogates exceeding the quality control limit and a positive results for bis(2-ethylhexyl)phthalate. The bis(2-ethylhexyl)phthalate results for these samples should be considered estimated biased high.

All front half and back half blank spiked samples reported recoveries within the quality control criteria. The condensate blank spike reported several recoveries outside the laboratory specified control limits but not the project required control limits.

#### 8.1.7 Blanks

<u>Requirements</u>: Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit.

Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below detection limit.

<u>Findings</u>: Blank train samples, reagent blanks, media blanks, and method blanks were analyzed as required. Bis(2-ethylhexy)phthalate) was reported in the blank train condensate sample and at equivalent levels in each of the condensate field samples. The condensate results for bis(2-ethylhexly)phthalate should be considered estimated biased high.

#### 8.1.8 Qualitative and Quantitative Results

Requiements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

Compounds were reported with a "J" qualifier by the laboratory. This qualifier indicates that the sample concentration was greater than the method detection limit and less than the reporting limit. Results "J" qualified by the laboratory should be considered estimated.

## 8.2 SIGNIFICANT FINDINGS

There were 2051 quality control criteria evaluated for this analysis. 2010 of the criteria were found to meet the project objectives and 41 were found to be outside control limits.

The 40% RPD of spike recoveries between surrogate requirement was not met by the front half samples because the Run 1 sample reported low recoveries for the surrogates. The RPDs between the Run 2 and Run 3 front half samples when compared without this sample were within the quality criteria. The Run 1 sample only reported one positive result, bis(2-ethylhexyl)phthalate. This sample result should be considered estimated. The condensate surrogates also did not meet this requirement. Positive results were reported for bis(2-ethylhexyl)phthalate in the condensate field samples. These results should be qualified estimated.

There were no duplicate injections of field samples performed. There was however blank spikes analyzed in duplicate of each matrix. The front and back half spiked reported RPDs within the quality control criteria. The condensate blank spike reported the 2,4-dimethylphenol RPD (64) exceeding the quality control criteria. There were no positive results for this compound, there for no qualification is required.

All front half and back half samples reported surrogate recoveries within the quality control criteria except G-2919/2920-R1-FH. Sample G-2919/2920-R1-FH reported four of the six surrogates below the control limit. Results for this sample should be considered estimated biased low. All of the condensate samples reported at least one base/neutral surrogate exceeding the quality control limit. Samples G-2923/2924-R1-CON, and G-3107/3108-R3-CON reported two bas/neutral surrogates exceeding the quality control limit and a positive results for bis(2-ethylhexyl)phthalate. The bis(2-ethylhexyl)phthalate results for these samples should be considered estimated biased high.

Blank train samples, reagent blanks, media blanks, and method blanks were analyzed as required. Bis(2-ethylhexy)phthalate) was reported in the blank train condensate sample and at equivalent levels in each of the condensate field samples. The condensate results for bis(2-ethylhexly)phthalate should be considered estimated biased high.

#### 9.0 ORGANOCHLORINE PESTICIDES

Stack gas organochlorine pesticides data were received in a single package. Samples G-2919/2930-R1-FH, G-2921/2922-R1-BH, G-2923/2924-R1-CON, G-3017/3018-R2-FH, G-2019/3020-R2-BH, G-3021/3022-R2-CON, G-3023/3024-R2-FH-BT, G-3025/3026-R2-BH-BT, G-3027/3028-R2-CON-BT, G-3029-R2-XAD-RB, G-3103/3104-R3-FH, G-3105/3106-R3-BH, and G-3107/3108-R3-CON were included. These samples were analyzed using SW846 Method 8081. All applicable compliance areas were reviewed and the significant findings discussed below. Section 9.1 provides a list of the primary data quality objectives evaluated during this review. Section 9.2 summarizes the significant findings of the evaluation.

## 9.1 DATA QUALITY OBJECTIVES

# 9.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

## 9.1.2 Sample Handling Criteria

<u>Requirements:</u> Samples must be chilled. Samples are to be held no more than 14 days from sampling to extraction and no more than 40 days from extraction to analysis.

<u>Findings</u>: All samples were chilled as required, extracted within 14 days, and analyzed within 40 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

## 9.1.3 Instrument Performance Criteria

<u>Requirement:</u> Monitor retention time windows. Retention times should be within 30 seconds of the last calibration check.

Findings: Retention times of reported compounds within the required retention time window.

## 9.1.4 Initial and Continuing Calibrations

Requirements: Five standards bracketing the expected concentration should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF should not exceed  $\pm$  15%.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each 12-hour shift. The continuing calibration response factors should be within  $\pm$  20% of the initial calibration mean RRF.

<u>Findings:</u> All initial calibration %RSDs less than 20% except DDD & Chlorobenzilate on column one, endosulfan sulfate on column two, and DCB on column two. The initial calibrations for these compounds were evaluated using correlation coefficients. All correlation coefficients were greater than 0.990. All continuing calibration response factors within 15% or the initial calibration

## 9.1.5 Precision Objectives

<u>Requirements:</u> <40% RPD (or 35% RSD, if greater than 4 determinations are made) of spike recoveries between field samples.

<35% RPD for duplicate injections from one run. This criterion is relaxed to 100% RPD if the compound is found at a concentration below the lowest standard.

<u>Findings:</u> All RPDs between field sample surrogate recoveries were within the quality control criteria. There was no duplicate injection of a field sample analyzed however, duplicate blank spikes analyzed for each matrix. All RPDs for the blank spike duplicate pairs were within the quality criteria.

## 9.1.6 Accuracy Objectives

<u>Requirements</u>: Recovery of isotopically labeled surrogates spiked into each sample as listed in Table 5-2 of the QAPP. If this criterion cannot be met, then the recoveries should be within 3 standard deviations of the historical means of surrogate recoveries for the method (laboratory specific).

Recovery of surrogates spiked onto a blank XAD-2 resin trap in the laboratory as listed in Table 5-2 of the QAPP.

<u>Findings</u>: All field samples reported surrogate recoveries within the quality criteria. All blank spiked samples reported target compound recoveries within the quality criteria except recoveries for delta-BHC (138%), gamma-BHC (136%), 4,4'-DDE (136%), and methoxychlor (138%) recoveries found in the blank spike associated with the front half/filter samples. The blank spike duplicate associated with these samples reported recoveries within the quality criteria for these compounds. There were no positive results for these compounds reported in the front half filter samples therefore no qualification is required.

# 9.1.7 Blanks

<u>Requirements</u>: Once during each test, a blank train is set up in the field and recovered like other field samples. Analysis of the blank train should be less than 20% of the sample levels or below the detection limit.

Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be less than 20% of sample levels or below detection limit.

<u>Findings:</u> The blank train, reagent blanks, media checks, and method blanks were analyzed as required. There were a few positive results reported in the blanks. These results were compared to the sample results. All positive sample results less than five times the highest associated blank should be qualified as estimated biased high. Below are lists of the positive blank results and of the field sample result reported less than five times the blank concentration.

Positive Blank Results

G-3023/3024-R2-FH-BT G-3025/3026-R2-BH-BT

Endrin aldehyde 0.019 J COL gamma-Chlordane 0.048 J COL

Heptachlor 0.034 J B COL

G-3027/3028-R2-CON-BT G-3029-R2-XAD-RB

 beta-BHC
 0.070 J COL
 4,4'-DDT
 0.037 J COL

 delta-BHC
 0.064 J COL
 Heptachlor
 0.014 J COL

Endosulfan II 0.020 J
Endrin aldehyde 0.11 B COL
Heptachlor 0.045 J COL
Heptachlor epoxide 0.020 J COL

Method Blank (condensate)

Method Blank (FH/Filter)

Endrin aldehyde 0.010 J Heptachlor 0.027 J COL

Field Samples With Results Less Than Five Times The Blank Concentration

G-2923/2924-R1-CON G-3021/3022-R2-CON G-3107/3108-R3-CON

Endrin aldehyde beta-BHC beta-BHC
Heptachlor delta-BHC delta-BHC
Endrin aldehyde Endrin aldehyde
Heptachlor Heptachlor

Heptachlor epoxide Heptachlor epoxide

## 9.1.8 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

Compounds were reported with a "COL" qualifier by the laboratory. This qualifier indicates that there was more than 40% difference in the concentrations between the primary and confirmation column. The laboratory SOP for this analysis requires that the lower of the two concentrations be reported in most cases because the lower result is considered better because the higher result is generally higher because of chromatographic interference. Results "COL" qualified by the laboratory should be considered estimated.

Compounds were reported with a "J" qualifier by the laboratory. This qualifier indicates that the sample concentration was greater than the method detection limit and less than the reporting limit. Results "J" qualified by the laboratory should be considered estimated.

# 9.2 SIGNIFICANT FINDINGS

There were 508 quality control criteria evaluated for this analysis. 493 of the criteria were found to meet the project objectives and 15 were found to be outside control limits.

The blank train, reagent blanks, media checks, and method blanks were analyzed as required. There were a few positive results reported in the blanks. These results were compared to the sample results. All positive sample results less than five times the highest associated blank should be qualified as

estimated biased high. Lists of the positive blank results and of the field sample result reported less than five times the blank concentration are included in section 9.1.7.

Compounds were reported with a "COL" qualifier by the laboratory. This qualifier indicates that there was more than 40% difference in the concentrations between the primary and confirmation column. The laboratory SOP for this analysis requires that the lower of the two concentrations be reported in most cases because the lower result is considered better because the higher result is generally higher because of chromatographic interference. Results "COL" qualified by the laboratory should be considered estimated.

#### 10.0 POLYCHLORINATED BIPHENYLS

Stack gas polychlorinated biphenyl data were received in a single package. Samples G-2925/2926-R1-FH, G-2927/2928-R1-BH, G-2929/2930-R1-CON, G-3030/3031-R2-FH, G-3032/3033-R2-BH, G-3034/3035-R2-CON, G-3036/3037-R2-FH-BT, G-3038/3039-R2-BH-BT, G-3040/3041-R2-CON-BT, G-3042-R2-XAD-RB, G-3109/3110-R3-FH, G3111/3112-R3-BH, and G-3113/3114-R3-CON were included. These samples were analyzed using EPA Method 1668. Section 10.1 provides a list of the primary data quality objectives evaluated during this review. Section 10.2 summarizes the significant findings of the evaluation.

### 10.1 DATA QUALITY OBJECTIVES

# 10.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings:</u> All sample reporting was provided as needed to confirm analytical results.

# 10.1.2 Sample Handling Criteria

<u>Requirements:</u> Samples must be chilled. Samples are to be held no more than 14 days from sampling to extraction and no more than 40 days from extraction to analysis.

<u>Findings:</u> All samples were chilled as required, extracted within 14 days, and analyzed within 40 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

# 10.1.3 Instrument Performance Criteria

<u>Requirements:</u> Retention time window verification and GC column performance should be checked at the beginning of each 12 hour shift and should meet the requirements of Section 15.4 of Method 1668.

<u>Findings:</u> Retention times monitored at the beginning of each 12 hour shift.

## 10.1.4 Initial and Continuing Calibrations

Requirements: Five high-resolution concentration calibration solutions should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF should not exceed ± 35%.

A midlevel standard should be run at the beginning of each 12-hour shift. The continuing calibration RFs should be within + 30%.

<u>Findings:</u> Initial and continuing calibrations were analyzed as required. All relative standard deviations, relative response factors and percent differences were within the required quality control criteria for reported compounds.

# 10.1.5 Precision Objectives

Requirements: <40% RPD (or 35% RSD, if greater than 4 determinations are made) of spike recoveries between field samples.

<35% RPD for duplicate injections from one run. This criterion is relaxed to 100% RPD if the compound is found at a concentration below the lowest standard.

<35% RPD for duplicate preparation and analysis of spiked blank XAD-2 resin trap. (Matrix Spike Duplicate)

<u>Findings:</u> All RPDs between internal standards and surrogates were less than the quality criteria. There was no duplicate injection of field samples performed however, there were blank spikes analyzed for each matrix. All of the blank spike RPDs were within the quality criteria.

## 10.1.6 Accuracy Objectives

<u>Requirements:</u> Recovery of isotopically labeled surrogates spiked into each sample as listed in Table 5-2 of the QAPP. If this criterion cannot be met, then the recoveries should be within 3 standard deviations of the historical means of surrogate recoveries for the method (laboratory specific).

Recovery of appropriate compounds spiked onto a blank XAD-2 resin trap in the laboratory as listed in Table 5-2 of the QAPP.

<u>Findings:</u> All surrogate recoveries were within limits. All field samples except G-3113/3114-R3-CON reported at least one internal standard exceeding the quality criteria however, none of the internal standards were associated with a positive sample result. All recoveries blank spike recoveries for each matrix were within the quality criteria. Internal standards reported exceeding the quality criteria are listed below.

# Internal Standards Exceeding the Quality Criteria

G-2925/2926-R1-FH 13C12-PCB 209	G-2927/2928-R1-BH 13C12-PCB 208 13C12-PCB 209	G-2929/2930-R1-CON 13C12-PCB 54 13C12-PCB 155 13C12-PCB 189
G-3030/3031-R2-FH 13C12-PCB 205 13C12-PCB 209	G-3032/3033-R2-BH 13C12-PCB 208 13C12-PCB 209	G-3034/3035-R2-CON 13C12-PCB 54 13C12-PCB 155 13C12-PCB 189
G-3109/3110-R3-FH 13C12-PCB 155 13C12-PCB 202 13C12-PCB 206 13C12-PCB 209	G-3111/3112-R3-BH 13C12-PCB 208 13C12-PCB 209	

### 10.1.7 Blanks

<u>Requirements:</u> Once during each test, a blank train is set up in the field and recovered in the same manner as other field samples. Analysis of the blank train is performed to assess contamination.

Analysis of one method blank for recovery reagents and XAD/filter, carried through all preparation and analysis steps, should be conducted to assess contamination

<u>Findings:</u> Blank trains, reagent blanks, media checks, and method blanks were analyzed as required. Bank trains, reagent blanks, media checks, and method blank reported positive results. The field sample result were compared to the blank results and results less than five times the highest associated blank concentration should be considered estimated blank concentration.

C 2020/2027 D2 FU DT	Positive Blank	Results G-3038/3039-R2-BH-BT	
G-3036/3037-R2-FH-BT Dichlorobiphenyl (total) Heptachlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Octachlorobiphenyl (total) Pentachlorobiphenyl (total) PCB 105 PCB 118 Tetrachlorobiphenyl (total) PCB 77 Trichlorobiphenyl (total)	4.0 QB 0.11 QJ 0.24 JQ 0.18 BJQ 0.037 J 0.66 JQB 0.037 QJ 0.093 J 2.3 QB 0.030 QJ 6.2 QB	Dichlorobiphenyl (total) Heptachlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Pentachlorobiphenyl (total) PCB 105 PCB 118 Tetrachlorobiphenyl (total) Trichlorobiphenyl (total)	0.60 QBJ 0.022 QJ 0.16 QBJ 0.032 J 0.29 JQB 0.024 QJ 0.046 QJ 1.1 QBJ 1.4 QJB
G-3040/3041-R2-Con-BT Dichlorobiphenyl (total) Heptachlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Octachlorobiphenyl (total) Pentachlorobiphenyl (total) PCB 105 PCB 118 Tetrachlorobiphenyl (total) PCB 77 Trichlorobiphenyl (total)	2.1 QB 0.022 QBJ 0.28 JQB 0.15 BJ 0.013 QJ 0.94 JQB 0.037 BJ 0.091 BJ 3.6 QB 0.024 QJ 5.7 BQ	G-3042-R2-XAD-RB Dichlorobiphenyl (total) Heptachlorobiphenyl (total) Hexachlorobiphenyl (total) Pentachlorobiphenyl (total) PCB 118 Tetrachlorobiphenyl (total) Trichlorobiphenyl (total)	0.15 QBJ 0.015 J 0.046 JQB 0.045 QJ 0.017 QJ 0.072 QBJ 0.12 BJQ
A-5376 Media Check XAD Dichlorobiphenyl (total) Heptachlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Pentachlorobiphenyl (total) Tetrachlorobiphenyl (total) Trichlorobiphenyl (total)	0.39 QBJ 0.011 QJ 0.042 QBJ 0.13 BJQ 0.065 QJB 0.30 BJQ 0.40 BJQ	A-5378 Media Check Filter Dichlorobiphenyl (total) Monochlorobiphenyl (total) Tetrachlorobiphenyl (total) Trichlorobiphenyl (total)	0.13 QBJ 0.045 BJ 0.077 QBJ 0.11 BJQ
Method Blank (Front Half/Filter) Dichlorobiphenyl (total) Monochlorobiphenyl (total) Pentachlorobiphenyl (total) Tetrachlorobiphenyl (total) Trichlorobiphenyl (total)	0.14 QJ 0.069 J 0.0097 QJ 0.086 QJ 0.15 QJ	Method Blank (Back Half/XAD) Dichlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Pentachlorobiphenyl (total) Tetrachlorobiphenyl (total) Trichlorbiphenyl (total)	0.61 QJ 0.059 QJ 0.13 JQ 0.034 J 0.18 QJ 0.33 JQ

Method Blank (Condensate)

Dichlorobiphenyl (total) 0.35 QJ Heptachlorobiphenyl (total) 0.010 JHexachlorobiphenyl (total) 0.027 QJ Monochlorobiphenyl (total) 0.11 QJ Pentachlorobiphenyl (total) 0.049 QJ **PCB 105** 0.0076 QJ **PCB 118** 0.012 J 0.0053 QJ **PCB 123** Tetrchlorobiphenyl (total) 0.25 QJ Trichlorobiphenyl (total) 0.36 QJ

# Field Samples Less Than Five Times The Blank Concentration

G-2925/2926-R1-FH	G-2927/2928-R1-BH	G-2929/2930-R1-Con
Dichlorobiphenyl (total)	Hexachlorobiphenyl (total)	Dichlorobiphenyl (total)
Hexachlorobiphenyl	Pentachlorobiphenyl (total)	Heptachlorobiphenyl (total)
Monochlorobiphenyl	PCB 105	Hexachlorobiphenyl (total)
Pentachlorobiphenyl	Tetrachlorobiphenyl (total)	Monochlorobiphenyl (total)
PCB 105	1 , ( ,	Pentachlorobiphenyl (total)
DOD 440		DOD 405

PCB 118 PCB 105 Tetrachlorobiphenyl (total) **PCB 118** 

Tetrachlorobiphenyl (total) **PCB 77** 

Trichlorobiphenyl (total) **PCB 77** 

Trichlorobiphenyl (total)

G-3030/3031-R2-FH G-3032/3033-R2-BH G-3034/3035-R2-Con Dichlorobiphenyl (total) Heptachlorobiphenyl (total) Dichlorobiphenyl (total) Hexachlorobiphenyl (total) Heptachlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Monochlorobiphenyl (total) Hexachlorobiphenyl (total) Pentachlorobiphneyl (total) Pentachlorobiphenyl (total) Monochlorobiphenyl (total) **PCB 118 PCB 105** Octchlorobiphenyl (total) Tetrachlorobiphneyl (total) **PCB 118** Pentachlorobiphenyl (total)

Trichlorobiphenyl (total) Tetrachlorobiphenyl (total) PCB 105

Trichlorobiphenyl (total) **PCB 118** 

Tetrachlorobiphenyl (total) **PCB 77** 

Trichlorobiphenyl (total)

G-3109/3110-R3-FH G3111/3112-R3-BH G3113/3114-R3-Con Dichlorobiphenyl (total) Hexachlorobiphenyl (total) Dichlorobiphenyl (total) Hexachlorobiphenyl (total) Pentachlorobiphenyl (total) Hexachlorobiphenyl (total) Monochlorobiphenyl (total) Monochlorobiphenyl (total) **PCB 105** Pentachlorobiphenyl (total) Pentachlorobiphenyl (total) **PCB 118** 

**PCB 118** Tetrachlorobiphenyl (total) PCB 105 Tetrachlorobiphenyl (total) Trichlorobiphenyl (total) **PCB 118 PCB 77 PCB 123** 

Trichlorobiphenyl (total) Tetrachlorobiphenyl (total) Trichlorobiphenyl (total)

#### 10.1.8 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings</u>: There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

Compounds qualified with a "C" qualifier by the laboratory indicate that the isomer is known to coelute with another member of the homologue group, or that the peak shape is shouldered, indicating the likelihood of a coeluting isomer. When a number follows the "C" flag, the number indicates the lowest numbered congener among the coelution set. Isomers qualified with a "C" by the laboratory should be considered estimated.

Compounds qualified with a "Q" by the laboratory indicate that the result is a maximum possible concentration. This qualifier is used when the result is generated from chromatographic data that does not meet all the qualitative criteria for a positive identification given in the method and listed in the data package care narrative. Results qualified by the laboratory with a "Q" qualifier should be considered estimated.

Compounds qualified with a "J" qualifier by the laboratory indicate that the result was reported between the estimated detection limit (EDL) and below the reporting limit (estimated minimum level. Results qualified by the laboratory with a "J" qualifier should be considered estimated.

### 10.2 SIGNIFICANT FINDINGS

There were 946 quality control criteria evaluated for this analysis. 881 of the criteria were found to meet the project objectives and 65 were found to be outside control limits.

Blank trains, reagent blanks, media checks, and method blanks were analyzed as required. Bank trains, reagent blanks, media checks, and method blank reported positive results. The field sample result were compared to the blank results and results less than five times the highest associated blank concentration should be considered estimated biased high. Lists of the positive blank results and field sample results less than five times the blank concentration are included in section 10.1.7.

## 11.0 STACK GAS POLYNUCLEAR AROMATIC HYDROCARBONS

The polynuclear aromatic hydrocarbon (PAH) data were received as a single package. Samples G-2925/2926-R1-FH, G-2927/2928-R1-BH, G-2929/2930-R1-CON, G-3030/3031-R2-FH, G-3032/3033-R2-BH, G-3034/3035-R2-CON, G-3036/3037-R2-FH-BT, G-3038/3039-R2-BH-BT, G-3040/3041-R2-CON-BT, G-3042-R2-XAD-RB, G-3109/3110-R3-FH, G3111/3112-R3-BH, and G-3113/3114-R3-CON were included. Samples were analyzed using CARB Method 429. Section 11.1 provides a list of the primary data quality objectives evaluated during this review. Section 11.2 summarizes the significant findings of the evaluation.

### 11.1 DATA QUALITY OBJECTIVES

# 11.1.1 Contract Compliance Monitoring

<u>Requirements</u>: Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

## 11.1.2 Sample Handling Criteria

<u>Requirements</u>: Samples must be chilled. Samples are to be held no more than 14 days from sampling to extraction and no more than 40 days from extraction to analysis.

<u>Findings:</u> All samples were chilled as required, extracted within 14 days, and analyzed within 40 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

### 11.1.3 Instrument Performance Criteria

Requirements: Retention time window within 30 seconds of the most recent calibration check verification and GC column performance should be checked at the beginning of each 12-hour shift.

<u>Findings</u>: Retention times and column performance check at the beginning of each 12-hour shift. Retention time requirements met.

# 11.1.4 Initial and Continuing Calibrations

Requirements: Five standards bracketing the expected concentration should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF for the labeled and unlabeled standards should not exceed  $\pm$  30% for both unlabeled analytes and internal standards.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each 12-hour shift. The continuing calibration RFs for all analytes should be within  $\pm$  30% of the initial calibration mean RRF.

<u>Findings:</u> Initial and continuing calibrations analyzed as required. All percent relative standards deviations and percent differences less than 30%.

## 11.1.5 Precision Objectives

Requirements: < 30% RSD of spike recoveries between samples for labeled compounds spiked prior to sampling.

- < 35% RSD of spike recoveries between samples for internal quantitation standards.
- < 50% RPD of spike recoveries between two laboratory control samples analyzed with each analytical batch.

<u>Findings</u>: All surrogate recovery %RSDs and all laboratory control sample RPDs were within the quality criteria. The relative standard deviation between the back half field samples for perylene-d12 (77.5) exceeded the control criteria Results for the back half sample. Sample G-3111/3112-R3-BH reported a low recovery for this internal standard (7.4%). Only the result from sample G-3111/3112-R3-BH should be considered estimated for perylene.

# 11.1.6 Accuracy Objectives

Requirements: 50 – 150% recovery of isotopically labeled PAH compounds spiked onto each sorbent resin tube prior to sampling

- 50 150% recovery of isotopically labeled internal quantitation standards spiked onto train components prior to extraction.
- 50 150% of the true value for analysis of two laboratory control samples analyzed with each analytical batch.

<u>Findings:</u> All surrogate recoveries and laboratory control sample recoveries were within the quality criteria. The recovery for perylene-d12 (7.4%) for sample G-3111/3112-R3-BH was below the quality criteria. The perylene-d12 result for this sample should be qualified estimated biased low.

#### 11.1.7 Blanks

<u>Requirements</u>: Once during each test, a blank train is set up in the field and recovered in the same manner as other field samples. Analysis of the blank train is performed to assess contamination.

Analysis of one method blank for recovery reagents and XAD/filter carried through all preparation and analysis steps should be conducted with results less than the detection limit.

<u>Findings</u>: The blank train, reagent blank, media checks, and method blanks were analyzed as required. Positive results were reported for blanks and the field sample results were compared to the blank concentrations and results less than five times the highest associated blank result should be considered estimated biased high. Below are lists of the positive blank results and field sample results less than five times the blank concentration.

# Positive Blank Results

G3036/3037-R2-FH-BT Acenaphthene Acenaphthalene Anthracene Benzo(a)anthracene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(ghi)perylene Benzo(a)pyrene Benzo(e)pyrene Chrysene Fluoranthene Fluoranthene Fluorene Indeno(123cd)pyrene 2-Methylnaphthalene Naphthalene Perylene Phenanthrene Pyrene	1.9 BJ 2.3 J 6.0 J 4.5 BJ 13 BJ 11 BJ 11 BJ 8.0 BJ 8.4 BJ 13 BJ 36 B 5.8 BJ 11 J 15 BJ 28 BJ 3.2 BJ 68 B 28 BJ	G-3038/3039-R2-BH-BT Acenaphthene Acenaphthalene Benzo(b)fluoranthene Benzo(ghi)perylene Chrysene Fluoranthene Fluoranthene Indeno(123cd)pyrene 2-Methylnaphthalene Naphthalene Perylene Phenanthrene Pyrene	4.2 BJ 1.3 J 39 B 3.5 J 3.3 J 2.4 J 9.6 BJ 2.8 BJ 1.4 J 17 BJ 200 BJ 1.6 BJ 12 B 19 BJ
G-3040/3041-R2-Con-BT Acenaphthene Acenaphthalene Anthracene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(ghi)perylene Benzo(a)pyrene Benzo(e)pyrene Chrysene Fluoranthene Fluorene 2-Methylnaphthalene Naphthalene Phenanthrene Pyrene	1.4 J 1.9 J 3.9 J 4.8 J 9.9 J 13 BJ 3.5 BJ 5.0 BJ 4.4 BJ 26 B 3.3 J 12 BJ 24 BJ 51 B 19 BJ	G-3042-R2-XAD-RB Anthracene Benzo(b)fluoranthene Benzo(ghi)perylene Fluoranthene Fluorene Indeno(123cd)pyrene 2-Methylnaphthalene Naphthalene Phenanthrene Pyrene	0.50 J 41 B 20 J 1.2 BJ 2.3 BJ 1.9 J 13 BJ 180 BJ 5.5 BJ 1.4 BJ
Method Blank (Front Half/Filter) Acenaphthene Benzo(a)anthracene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Benzo(e)pyrene Chrysene Fluoranthene Fluorene Indeno(123cd)pyrene 2-Methylnaphthalene Naphthalene Perylene Phenanthrene Pyrene	4.2 J 1.6 J U 4.2 J 4.7 J 5.0 J 4.5 J 1.8 J 1.1 J 1.3 J 3.6 J 5.6 J 12 J 4.4 J 1.9 J 2.5 J	Method Blank (Back Half/XAD) Acenaphthene Benzo(b)fluoranthene Benzo(a)pyrene Benzo(e)pyrene Fluoranthene Fluoranthene Fluorene 2-Methylnaphthalene Naphthalene Perylene Phenanthrene Pyrene	3.1 J 3.9 J 5.2 J 4.0 J 1.6 J 3.0 J 13 J 23 J 5.2 J 5.7 J 1.7 J

A-5376 Media Check XAD		A-5378 Media Check Filter	
Benzo(b)fluoranthene	33 B	Benzo(a)anthracene	0.98 BJ
		Benzo(b)fluoranthene	3.2 BJ
		Benzo(k)fluoranthene	2.5 BJ
		Chrysene	1.1 BJ
		Indeno(123cd)pyrene	3.1 BJ

Method Blank (Condensate) Benzo(a)anthracene 2.3 J Benzo(ghi)perylene 6.0 J Benzo(a)pyrene 8.2 J Benzo(e)pyrene 8.4 J Chrysene 2.8 J Fluoranthene 1.3 J Indeno(123cd)pyrene 6.9 J 2-Methylnaphthalene 8.6 J Naphthalene 22 J Perylene 8.3 J Phenanthrene 2.4 J Pyrene 1.5 J

# Field Sample Results Less Than five Times Blank Concentration

Anthracene Benzo(a)anthracene Benzo(b)fluoranthene Benzo(a)anthracene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(b)fluoranthene Benzo(ghi)perylene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(ghi)perylene Benzo(ghi)perylene Fluorene Benzo(a)pyrene Indeno(123cd)pyrene Chrrsene Indeno(123cd)pyrene Chrysene Naphthalene Fluorene Indeno(123cd)pyrene Indeno(123cd)pyrene 2-Methylnaphthalene Perylene Indeno(123cd)pyrene 2-Methylnaphthalene Phee	Anthracene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(ghi)perylene Benzo(a)pyrene Benzo(e)pyrene Chrysene Fluoranthene Fluoranthene Fluorene ndeno(123cd)pyrene 2-Methylnaphthalene Naphthalene Phenanthrene
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G-3030/3031-R2-FH G-3032/3033-R2-BH G3034/3035-R2 Acenaphthene Acenaphthene Acenaphthene Fluoranthene Acenaphthalene Anthracene Fluorene Benzo(b)fluoranthene Benzo(b)fluoranthene 2-Methylnaphthalene Benzo(k)fluoranthene Benzo(ghi)perylene Naphthalene Benzo(ghi)perylene Benzo(a)pyrene Phenanthrene Fluoranthene Benzo(e)pyrene Pyrene

ridorantherie Fluorene Chrysene

2-Methylnaphthalene Fluorene

Pyrene Fluorene

Indeno(123cd)pyrene 2-Methylnaphthalene Naphthalene Phenanthrene

Pyrene

G-3109/3110-R3-FH Acenaphthene Benzo(b)fluoranthene Benzo(ghi)perylene Benzo(a)pyrene Benzo(e)pyrene Fluoranthene Fluorene

Indeno(123cd)pyrene 2-Methylnaphthalene Naphthalene

Phenanthrene Pyrene

G-3111/3112-R3-BH Acenaphthene Acenaphthalene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(ghi)perylene Benzo(e)pyrene Chrysene Fluoranthene Fluorene

2-Methylnaphthalene

Indeno(123cd)pyrene

Pyrene

G3113/3114-R3-Con Benzo(b)fluoranthene Benzo(k)fluoranthene

Fluoranthene Fluorene

2-Methylnaphthalene

Naphthalene Phenanthrene Pyrene

# 11.1.8 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

There were no transcription errors found during the review of this data. There were no Findings: additional issues not already discussed noted.

Compounds qualified with a "J" qualifier by the laboratory indicate that the result was reported between the estimated detection limit (EDL) and below the reporting limit (estimated minimum level). Results qualified by the laboratory with a "J" qualifier should be considered estimated.

### 11.2 SIGNIFICANT FINDINGS

There were 716 quality control criteria evaluated for this analysis. 613 of the criteria were found to meet the project objectives and 103 were found to be outside control limits.

All surrogate recoveries and recovery %RSDs and all laboratory control sample recoveries and RPDs were within the quality criteria. The relative standard deviation between the back half field samples for perylene-d12 (77.5) exceeded the control criteria Results for the back half sample. Sample G-3111/3112-R3-BH reported a low recovery for this internal standard (7.4%). Only the result from sample G-3111/3112-R3-BH should be considered estimated biased low for pervlene.

Blank trains, reagent blanks, media checks, and method blanks were analyzed as required. Bank trains, reagent blanks, media checks, and method blank reported positive results. The field sample result were compared to the blank results and results less than five times the highest associated blank concentration should be considered estimated biased high. Lists of the positive blank results and field sample results less than five times the blank concentration are included in section 11.1.7.

#### 12.0 STACK GAS TOTAL SEMIVOLATILE AND NONVOLATILE ORGANICS

The stack gas total semivolatile data were received as a single package for both TCO and gravimetric analysis. Samples G-2931/2932-R1-FH, G-2933/2934-R1-BH, G-2935/2936-R1-CON, G-3043/3044-R2-FH, G3045/3046-R2-BH, G-3047/3048-R2-CON, G-3115/30116-R3-FH, G3117/3118-R3-BH, G-3119/3120-R3-CON, G-3121/3122-R3-FH-BT, G-3123/3124-R3-BH-BT, G-3125/3126-R3-CON-BT, and G-3127-R3-XAD-RB were included. The samples were analyzed using GC/FID and gravimetrically. All applicable compliance areas were reviewed and the significant findings discussed below. Section 12.1 provides a list of the primary data quality objectives evaluated during this review. Section 12.2 summarizes the significant findings of the evaluation.

## 12.1 DATA QUALITY OBJECTIVES

## 12.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

# 12.1.2 Sample Handling Criteria

<u>Requirements</u>: Samples must be chilled. Samples are to be held no more than 14 days from sampling to extraction and no more than 40 days from extraction to analysis.

<u>Findings:</u> All samples were chilled as required, extracted within 14 days, and analyzed within 40 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

# 12.1.3 Initial and Continuing Calibrations (TCO)

<u>Requirements:</u> TCO - Three to five standards bracketing the expected concentration should be used for the initial calibration.

TCO - Initial calibration correlation coefficient ≥ 0.97

TCO - A midlevel standard should be run daily and be within + 15% of the true value.

<u>Findings</u>: The TCO initial calibration contained 6 standards. All correlation coefficients were greater than 0.97. A midlevel standard was analyzed prior to analysis on the day our samples were run. The recovery of the midlevel standard was within + 15% of the true value,

# 12.1.4 Precision Objectives

Requirements: < 15% RPD between the analysis of one replicate sample for TOC per test.

< 20% RPD between duplicate analysis of each sample for the gravimetric analysis.

<u>Findings:</u> Duplicates were not analyzed for any of the field samples. There were blank spike duplicate analyzed for each matrix for both the TCO and gravimetric analysis. All RPDs for these spiked samples were within the quality criteria.

## 12.1.5 Accuracy Objectives

Requirements: TCO - A midlevel standard should be run daily and be within ± 15% of the true value.

Grav audit sample + 20% of actual value

<u>Findings:</u> The TCO midlevel standard reported a value within  $\pm$  15% of the true value. There were four gravimetric audit samples analyzed reporting results all within  $\pm$  20 of the actual value. Additionally laboratory control samples were analyzed for both analyses and surrogates were included for the TCO analysis with good recoveries reported.

#### 12.1.6 Blanks

Requirements: Analysis of one method blank per sample batch carried through all preparation and analysis steps should be less than 20% of sample levels or below the detection limit.

Once during the test a blank train is set up and recovered like other field samples. The results of this train are used to assess contamination.

Both the TCO and GRAV analysis may be blank corrected as allowed by the method.

<u>Findings:</u> The blank train, reagent blank, media checks, and method blanks were analyzed as required. Positive results were reported for blanks and the field sample results were compared to the blank concentrations and results less than five times the highest associated blank result should be considered estimated biased high. Below are lists of the positive blank results and field sample results less than five times the blank concentration.

## Positive Blank Results

G-3121/3122-F	R3-FH-BT	G-3123/3124-R3-BH-BT		G-3125/3126-R3-CON-BT	
TCO	0.022 mg	TCO	1.1 B mg	TCO	0.49 B
Grav	0.33 JB mg	Grav	1.0 B mg		
G-3127-R3-XA	D-RB	Filter Media Ch	neck		
TCO	0.15 B mg	TCO	0.0060 J mg		
	Ū	Grav	0.33 JB mg		
Method Blank (	(EU/Eiltor)	Method Blank	(DU/V \ D)	Method Blank	Condonasta
,			'		
Grav	0.40 J mg	TCO	0.0022 J mg	TCO	0.019 J mg
		Grav	0.33 J mg		

Field Sample Results Less Than Five Times The Blank Concentration

G-2931/2932-R1-FH	G-2933/2934-R1-BH	G-2935/2936-R1-CON
Grav	TCO	TCO
	Grav	

G-3043/3044-R2-FH G-3045/3046-R2-BH G-3047/3048-R2-CON

Grav TCO TCO

Grav

G-3115/3116-R3-FH G-3117/3118-R3-BH G-3119/3120-R3-CON

TCO TCO TCO

Grav Grav

#### 12.1.7 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

Compounds qualified with a "J" qualifier by the laboratory indicate that the result was reported between the method detection limit (MDL) and below the reporting limit. Results qualified by the laboratory with a "J" qualifier should be considered estimated.

The total semivolatile and nonvolatile organic results were blank qualified using the method blanks. Also, the laboratory noted that they believed that some of the Run three samples were switched with the blank train samples because the condensate and front half TCO results for the blank train were more like the other runs results and Run 3 reported very low TCO results for the front half and condensate samples. A complete review of the results does not provide convincing evidence either for or against this theory. The results should be used as reported.

# 12.2 SIGNIFICANT FINDINGS

There were 32 quality control criteria evaluated for this analysis. 20 of the criteria were found to meet the project objectives and 12 were found to be outside control limits.

The blank train, reagent blank, media checks, and method blanks were analyzed as required. Positive results were reported for blanks and the field sample results were compared to the blank concentrations and results less than five times the highest associated blank result should be considered estimated biased high. Lists of the positive blank results and field sample results less than five times the blank concentration are included in section 12.1.7.

#### 13.0 STACK GAS HEXAVALENT CHROMIUM

The stack gas hexavalent chromium data were received as a single package. Samples G-2944-R1, G-2945-R1-FS, G-2946-R1-FSD, G-2947-R1-RB, G-2948-R1-RBS, G-3054-R2, and G-3138-R3 were included. The samples were analyzed for hexavalent chromium by ion chromatography SW846 Method 7199. Applicable compliance areas were reviewed and the significant findings discussed below. Section 13.1 provides a list of the primary data quality objectives evaluated during this review. Section 12.2 summarizes the significant findings of the evaluation.

### 13.1 DATA QUALITY OBJECTIVES

# 13.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings</u>: All sample reporting was provided as needed to confirm analytical results.

The initial calibration for this analysis did not have the calibration standards analyzed in duplicate. Six standards were analyzed and the correlation coefficient calculated to determine linearity. The correlation coefficient for the initial calibration was 0.9986.

# 13.1.2 Sample Handling Criteria

Requirements: Samples must be chilled with pH > 8.5. Samples are to be held no more than 14 days.

<u>Findings:</u> All samples were chilled and preserved as required and analyzed within 14 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

# 13.1.3 Initial and Continuing Calibrations

Requirements: IC - Four standards bracketing the expected concentration should be used for the initial calibration. RPD of responses of the two standards at each concentration < 10% with an accuracy of  $\pm$  7%

IC- Calibration check to be analyzed one per test with an accuracy of  $\pm$  10%

<u>Findings:</u> The initial calibration for this analysis did not have the calibration standards analyzed in duplicate. Six standards were analyzed and the correlation coefficient calculated to determine linearity. The correlation coefficient for the initial calibration was 0.9986. Three calibration checks were analyzed with these samples. All of the percent recoveries were within the quality criteria.

## 13.1.4 Precision Objectives

Requirements: The RPD of duplicate analysis should be < 30%.

<u>Findings</u>: All field samples and field spikes were analyzed in duplicate. All RPDs were within the quality criteria.

## 13.1.5 Accuracy Objectives

Requirements: Recovery of hexavalent chromium spiked onto one sample preparation should be 60 – 140%.

<u>Findings:</u> Recovery for the matrix spike / matrix spike duplicate performed on the run one sample were within the quality criteria. Additionally two field spikes of the run one sample at two different levels and a field spike of the reagent blank were prepared in the field and sent to the laboratory for analysis with the field samples to confirm adequate preservation of these samples. All of the recoveries for these samples were between 86.5% and 96.3%.

#### 13.1.6 Blanks

<u>Requirements:</u> Analysis of one set of reagent blanks and a method blank carried through all preparation and analysis steps, should be less than 20% of sample levels or below the detection limit.

<u>Findings</u>: Reagent blanks and method blanks were analyzed as required. There were no positive results reported for any blank.

### 13.1.7 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

# 13.2 SIGNIFICANT FINDINGS

There were 27 quality control criteria evaluated for this analysis. 27 of the criteria were found to meet the project objectives and 0 were found to be outside control limits.

#### 14.0 PROCESS VOLATILE ORGANICS

The feed and process volatile organics data were received in a single data package. Samples G-2889-R1-Carbon, G-2893-R1-Scrubber, G-2897-R1-POTW, G-2901-R1-Caustic, G-2905-R1-Water, G-2987-R2-Carbon, G-2991-R2-Scrubber, G-2995-R2-POTW, G-3003-R2-Water, G-3070-R3-Carbon, G-3074-R3-Scrubber, G-3078-R3-POTW, and G-3086-R3-Water were included and analyzed by GC/MS using SW-846 method 8260. Applicable compliance areas were reviewed and the significant findings discussed below. Section 14.1 provides a list of the primary data quality objectives evaluated during this review. Section 14.2 summarizes the significant findings of the evaluation.

### 14.1 DATA QUALITY OBJECTIVES

# 14.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings:</u> All sample reporting was provided as needed to confirm analytical results.

# 14.1.2 Sample Handling Criteria

Requirements: Samples must be chilled. Samples are to be held no more than 14 days.

<u>Findings</u>: All samples were chilled as required and analyzed within 14 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

## 14.1.3 Instrument Performance Criteria

<u>Requirements</u>: Internal standard spiked into each sample, standard and, blank should report a retention time within 30 seconds and an area within – 50 to + 100% of the last calibration check.

<u>Findings</u>: All internal standards were within the quality criteria for both retention time and area for all samples.

## 14.1.4 Initial and Continuing Calibrations

Requirements: Three to five standards bracketing the expected concentration should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF for the CCCs should not exceed  $\pm$  30% and the mean RRF for the SPCC compounds should be > 0.3 for chlorobenzene and 1,1,2,2-tetrachloroethane and > 0.1 for chloromethane, 1,1-dichloroethane, and bromoform.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each 12-hour shift. The continuing calibration RFs should be within  $\pm$  25% of the initial calibration mean RRF for CCCs. The SPCCs should also be > 0.3 for chlorobenzene and 1,1,2,2-tetrachloroethane and > 0.1 for chloromethane, 1,1-dichloroethane, and bromoform.

Findings: All CCCs and SPCCs met the quality criteria in both the initial and continuing calibration. It was noted that the compounds listed below were not within 25% of the initial calibration mean RRF in the given continuing calibration. The only positive result associated with one of these compounds is the iodomethane result for G-2889-R1-Carbon. This result should be qualified as estimated.

Continuing Calibration Compounds Greater Than 25% Difference From The Initial Calibration Mean RRF

Continuing Calibration 4/4/06 Trichlorofluoromethane Iodomethane 2,2-Dichloropropane	45.0 50.5 70.0
Continuing Calibration 4/5/06 Bromomethane Carbon tetrachloride 1,1,1,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane	26.2 29.3 25.1 34.5
Continuing Calibration 4/6/06 trans-1,3-Dichloropropene 1,2-Dibromo-3-chloropropane	27.1 33.0

# 14.1.5 Precision Objectives

 $\frac{\text{Requirements:}}{\text{RPD between duplicate preparations of one feed sample and /or} < 35\% \text{ RPD for the matrix spike/matrix spike duplicate analysis (MS/MSD)}.$ 

<u>Findings:</u> A matrix spike / matrix spike duplicate was performed on one sample for each matrix. Below is a list of the RPDs reported exceeding the quality criteria. Not included in this list are samples with zero percent recovery for one spiked sample as RPDs are not valid. The only positive sample results associated with the high MS/MSD RPDs were the toluene results from the three spent activated carbon samples, G-2889-R1-Carbon, G-2987-R2-Carbon, G-3070-R3-Carbon. These sample results should be qualified as estimated.

## MS/MSD RPDs Exceeding the Quality Criteria

	Run 1 Scrubber MS/MSD	
35	n-Butylbenzene	57
152	sec-Butylbenzene	44
44	tert-Butylbenzene	36
47	2-Chlorotoluene	47
37	4-Chlorotoluene	49
	1,2-Dichlorobenzene	50
	1,3-Dichlorobenzene	52
68	1,4-Dichlorobenzene	55
	Hexachlorobutadiene	50
	p-Isopropyltoluene	45
64	•	57
	n-Propylbenzene	44
	Styrene	43
	1,2,3-Trichlorobenzene	53
		46
	•	53
	1,3,5-trimethylbenzene	47
	152 44 47 37 68	35 n-Butylbenzene 152 sec-Butylbenzene 44 tert-Butylbenzene 47 2-Chlorotoluene 37 4-Chlorotoluene 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Hexachlorobutadiene p-Isopropyltoluene 64 Naphthalene n-Propylbenzene Styrene

# 14.1.6 Accuracy Objectives

Requirements: 50 – 130% recovery of standards spiked onto a sample (matrix spike)

50 - 130% recovery of isotopically labeled POHCs or appropriate surrogates spiked onto every field sample.

<u>Findings</u>: High surrogate recoveries were reported for sample G-2901-R1-Caustic, caustic feed. Positive results were reported for acetone, bromobenzene, bromodichloromethane, bromoform, chlorodibromomethane, chloroform, 1,2-dichloroethane, methylene chloride, and trichloroethene. These results should be considered estimated biased high. Spent activated carbon samples, G-2889-R1-Carbon, G-2987-R1-Carbon, and G-3070-R3-Carbon, all reported extremely low surrogate recoveries as would be expected from this matrix. All of the results for these samples should be considered suspect and should be qualified as estimated biased low.

Laboratory control samples were analyzed in association with all samples and reported recoveries within the quality criteria except for the acetone recovery associated with samples G-2893-R1-Scrubber, G-2991-R2-Scrubber, G-2995-R2-POTW, G-3003-R2-Water, G-3074-R3-Scrubber, G-3078-R3-POTW, and G-3086-R3-Water. This acetone recovery was reported slightly low (47%). The acetone results for these samples should be considered estimated biased low. Several of the matrix spike / matrix spike duplicate results reported low recoveries for the spent activated carbon samples and the scrubber water samples indicating a matrix effect. All compounds reporting low recoveries should be considered estimated biased low. Below is a list of the low recoveries for each matrix. Not included in this list are compounds with original results more than four times the spike concentration added.

# Low Matrix Spike / Matrix Spike duplicate Recoveries

Dura 4 Corbon MC/MCD	
Run 1 Carbon MS/MSD	00 MCD
Acetone	39 MSD
Acrylonitrile	31 / 34 0 / 0
Benzene Bromobenzene	0/0
Bromochloromethane	15 / 15
Bromodichloromethane	8.4 / 8.9
Bromoform	0/0
Bromomethane	4.7 / 14
n-Butylbenzene	0/0
sec-Butylbenzene	0/0
tert-Butylbenzene	0/0
Carbon disulfide	5.4 / 6.1
Carbon tetrachloride	7.8 / 9.5
Chlorobenzene	0/0
Chlorodibromomethane	0/0
Chloroethane	28 / 40
Chloroform	35 / 42
2-Chlorotoluene	0/0
4-chlorotoluene	0/0
1,2-Dibromo-3-chloropropane	5.3 / 0
1,2-Dibromomethane	0/0
Dibromomethane	0/0
1,2-Dichlorobenzene	0/0
1,3-Dichlorobenzene	0/0
1,4-Dichlorobenzene	0/0
Dichlorodifluoromethane	23 / 29
1,1-Dichloroethane	15 / 17
1,2-Dichloroethane	32 / 26
cis-1,2-Dichloroethene	7.7 / 13
trans-1,2-Dichloroethene	0 / 5.1
1,1-Dichloroethene	4.0 / 7.3
1,2-dichloropropane	8.6 MS
1,3-Dichloropropane	6.6 / 7.8
2,2-Dichloropropane	0/0
cis-1,3-Dichloropropene	0/0
trans-1,3-Dichloropropene	0 / 5.7
1,1-Dichloropropene	0 / 0 8.4 / 0
Ethylbenzene Hexachlorobutadiene	0/0
2-Hexanone	0/0
Isopropylbenzene	0/0
p-Isopropyltoluene	0/0
Methylene chloride	26 / 49
4-Methyl-2-pentanone	7.8 / 9.0
Naphthalene	8.5 / 0
n-Propylbenzene	0/0
Styrene	0/0
1,1,1,2-Tetrachloroethane	0/0
1,1,1,1-Tetrachloroethane	0/0
Tetrachloroethene	0/0
Toluene	9.9 / 1.3
1,2,3-Trichlorobenzene	0/0
1,2,4-Trichlorobenzene	0/0
1,1,1-Trichloroethane	18 / 6.5
1,1,2-Trichloroethane	15 / 10
Trichlorotrifluorormethane	25 / 33
1,2,3-Trichloropropane	0/0
1,2,4-trimethylbenzene	0/0
1,3,5-Trimethylbenzene	0/0
Vinyl chloride	21 / 27
m & p-Xylene	0/0
o-Xylene	0/0
Xylenes (total)	0 / 0)

Run 1 Scrubber Blowdown MS/MSD Bromobenzene	43 MSD
n-Butylbenzene	23 / 13
sec-Butyl benzene	41 / 26
tert-Butylbenzene	39 MSD
Chlorobenzene	48 MSD
2-Chlorotoluene	47 / 29
4-Chlorotoluene	42 / 26
1,2-Dichlorobenzene	44 / 27
1,3-Dichlorobenzene	39 / 23
1,4-Dichlorobenzene	38 / 22
Ethylbenzene	48 MSD
Hexachlorobutadiene	33 / 20
Isopropyltoluene	43 MSD
p-Isopropyltoluene	40 / 25
Naphthalene	13 / 7.5
n-Propylbenzene	43 / 27
Styrene	45 / 29
1,2,3-Trichlorobenzene	17 / 10
1,2,4-trichlorobenzene	15 / 9.6
1,2,4-Trimethylbenzene	38 / 22
1,3,5-trimethylbenzene	46 / 29
m & p-Xylene	40 MSD
o-Xylene	42 MSD
Xylenes (total)	41 MSD

#### 14.1.7 Blanks

<u>Requirements</u>: One method blank per batch carried through all preparation steps. Results should be less than the lowest standard.

<u>Findings</u>: Method blanks were analyzed as required. There were positive results reported in the all three method blanks. The field sample results were compared to the concentrations found in the blanks and all positive results less than five time the concentration found in the method blank should be considered estimated biased high. Below is a list of the method blank positive results. The only positive results associated with the positive method black results were the bromomethane results for samples G-2889-Carbon and G-2978-R2-Carbon and the lodomethane results for G-2889-R1-Carbon and G-2893-R1-Scrubber. These results should be considered estimated biased high.

## Positive Method Blank Results

Method Blank (Spent Activated Carbon)
Acetone 340 J
Bromomethane 260 J
Iodomethane 270 J

Method Blank (Scrubber Blowdown, POTW Runs 2 & 3, and Make-up Water Runs 2 & 3)

Iodomethane0.55 J1,2,3-Trichlorobenzene0.26 J1,2,4-trichlorobenzene0.15 J

Method Blank (Caustic Feed Run 1, Make-up Water Run 1, and POTW Run 1)

lodomethane 0.56 J

## 14.1.8 Qualitative and Quantitative Results

<u>Requirements</u>: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

#### 14.2 SIGNIFICANT FINDINGS

There were 1949 quality control criteria evaluated for this analysis. 1660 of the criteria were found to meet the project objectives and 289 were found to be outside control limits.

All CCCs and SPCCs met the quality criteria in both the initial and continuing calibration. It was noted that the compounds listed in section 14.1.4 were not within 25% of the initial calibration mean RRF in the given continuing calibration. The only positive result associated with one of these compounds is the iodomethane result for G-2889-R1-Carbon. This result should be qualified as estimated.

A matrix spike / matrix spike duplicate was performed on one sample for each matrix. A list of the RPDs reported exceeding the quality criteria was included in section 14.1.5. The only positive sample results associated with the high MS/MSD RPDs were the toluene results from the three spent activated carbon samples, G-2889-R1-Carbon, G-2987-R2-Carbon, G-3070-R3-Carbon. These sample results should be qualified as estimated.

High surrogate recoveries were reported for sample G-2901-R1-Caustic, caustic feed. Positive results were reported for acetone, bromobenzene, bromodichloromethane, bromoform, chlorodibromomethane, chloroform, 1,2-dichloroethane, methylene chloride, and trichloroethene. These results should be considered estimated biased high. Spent activated carbon samples, G-2889-R1-Carbon, G-2987-R1-Carbon, and G-3070-R3-Carbon, all reported extremely low surrogate recoveries as would be expected from this matrix. All of the results for these samples should be considered suspect and should be qualified as estimated biased low.

Laboratory control samples were analyzed in association with all samples and reported recoveries within the quality criteria except for the acetone recovery associated with samples G-2893-R1-Scrubber, G-2991-R2-Scrubber, G-2995-R2-POTW, G-3003-R2-Water, G-3074-R3-Scrubber, G-3078-R3-POTW, and G-3086-R3-Water. This acetone recovery was reported slightly low (47%). The acetone results for these samples should be considered estimated biased low. Several of the matrix spike / matrix spike duplicate results reported low recoveries for the spent activated carbon samples and the scrubber water samples indicating a matrix effect. All compounds reporting low recoveries should be considered estimated biased low. A list of the low recoveries for each matrix is provided in section 14.1.6.

Method blanks were analyzed as required. There were positive results reported in the all three method blanks. The field sample results were compared to the concentrations found in the blanks and all positive results less than five time the concentration found in the method blank should be considered estimated biased high. A list of the method blank positive results is provided in section 14.1.7. The only positive results associated with the positive method black results were the bromomethane results for samples G-2889-Carbon and G-2978-R2-Carbon and the lodomethane results for G-2889-R1-Carbon and G-2893-R1-Scrubber. These results should be considered estimated biased high.

#### 15.0 PROCESS SEMIVOLATILES

The results for the process semivolatile organics process data were received in a single data package feed were received as a single package. Samples G-2890-R1-Carbon, G-2894-R1-Scrubber, G-2898-R1-POTW, G-2902-R1-Caustic, G-2906-R1-Water, G-2988-R2-Carbon, G-2992-R2-Scrubber, G-2996-R2-POTW, G-3004-R2-Water, G-3071-R3-Carbon, G-3075-R3-Scrubber, G-3079-POTW, and G-3087-R3-Water were included in this package and analyzed using a SW846 Method 8270. All applicable compliance areas were reviewed and the significant findings discussed below. Section 15.1 provides a list of the primary data quality objectives evaluated during this review. Section 15.2 summarizes the significant findings of the evaluation.

### 15.1 DATA QUALITY OBJECTIVES

## 15.1.1 Contract Compliance Monitoring

<u>Requirements:</u> Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

<u>Findings:</u> All sample reporting was provided as needed to confirm analytical results.

## 15.1.2 Sample Handling Criteria

<u>Requirements</u>: Samples must be chilled. Samples are to be held no more than 14 days from sampling to extraction and no more than 40 days from extraction to analysis.

<u>Findings:</u> All samples were chilled as required, extracted within 14 days, and analyzed within 40 days. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

### 15.1.3 Instrument Performance Criteria

<u>Requirements:</u> Internal standard spiked into each sample, standard and, blank should report a retention time within 30 seconds and an area within – 50 to + 100% of the last calibration check.

<u>Findings</u>: All internal standards were within the quality criteria for both retention time and area for all samples.

## 15.1.4 Initial and Continuing Calibrations

<u>Requirements:</u> Three to five standards bracketing the expected concentration should be used for the initial calibration.

The initial calibration %RSDs for the mean RRF for the CCCs should not exceed  $\pm$  30% and SCPP RRFs should be > 0.05.

A midlevel standard should be run at the beginning and end of analysis or at the beginning of each 12-hour shift. The continuing calibration RFs should be within  $\pm$  30% of the initial calibration mean RRF for CCCs. SPCC RRFs should be > 0.05

<u>Findings</u>: All initial and continuing calibrations were within the quality criteria and all target compounds also met the quality criteria for initial calibration RSDs and continuing calibration percent difference.

# 15.1.5 Precision Objectives

<u>Requirements:</u> < 35% RPD between duplicate preparations of one feed sample and /or < 35% RPD for the matrix spike/matrix spike duplicate analysis (MS/MSD).

<u>Findings:</u> A matrix spike / matrix spike duplicate was performed on one sample for each matrix. Below is a list of the RPDs reported exceeding the quality criteria. Not included in this list are samples with zero percent recovery for one spiked sample as RPDs are not valid. There were no positive results associated with any compound reporting a high RPD therefore no qualification is needed.

# Compounds Reporting High MS/MSD RPDs

Spent Activated Carbon MS/MSD					
4-Nitrophenol	38				
Pentachlorophenol	70				
- Chachierophener	, 0				
Caustic Feed MS/MAD					
Benzoic Acid	180				
4,6-Dinitro-2-methylphenol	191				
2-Nitrophenol	156				
4-Nitrophenol	187				
Pentachlorophenol	156				
2,4,5-Trichlorophenol	45				
2,4,6-Trichlorophenol	68				
Make-up Water MS/MSD					
Aniline	43				

## 15.1.6 Accuracy Objectives

Requirements: Recovery of appropriate surrogates spiked into each sample as specified in Table 5-2 of the QAPP

Matrix spike recovery within limits specified in Table 5-2 of the QAPP or laboratory specific limits.

<u>Findings</u>: All three spent activated carbon samples reported the 2,4,6-tribromophenol and the terphentl-d14 surrogates below the quality criteria. Since these were one surrogate from the acid fraction of the analysis and one surrogate from the base/neutral fraction of the analysis and all other surrogates were reported within the quality criteria, no qualification is needed. The make-up water samples G-3004-R2-Water and G-3087-Water both reported the 2-fluorophenol (3.0% & 0.87%)and phenol-d5 (0.41% & 0.0%) surrogates below the quality criteria. Both of these surrogates are from the acid fraction of the

sample. There was no sufficient sample volume for re-extraction and reanalysis of these samples. All results from these samples should be considered suspect and qualified estimated biased low.

Laboratory control samples were analyzed in association with these samples with all recoveries within the quality criteria. Matrix spike / matrix spike duplicate samples were analyzed for each matrix. There were several recoveries reported outside the quality criteria indicating a matrix effect. Below is a list of the recoveries reported below the criteria. All of the results for compounds for the same matrix should be qualified as estimated biased low for recoveries below the criteria. Only the 2-methylnaphthalene and naphthalene recoveries for the spent activated carbon matrix reported high recoveries when the original result was not more than four times the spike. All three carbon samples reported positive results for these compounds and should be considered estimated biased high.

# MS/MSD Recoveries Reported Below The Quality Criteria

Run 1 Spent Activated Carbon MS/MSD	)
Acenaphthene	39 MS
Acenaphthylene	35 MS
Anthracene	14 / 21
Benz(a)anthracene	3.7 / 4.8
Benzo(b)fluoranthene	0 / 2.7
Benzo(k)fluoranthene	0 / 2.0
Benzoic acid	17 MS
Benzo(ghi)perylene	0/0
Benzo(a)pyrene	0/3.2
Carbazole	25 / 33
2-Chloronaphthalene	29 / 37
Chrysene	3.7 / 5.1
Dibenz(ah)anthracene	0/0
Dibenzofuran	24 / 32
3,3'-Dichlorobenzidine	15 MS
4,6-Dinitro-2-methylphenol	7.3 / 8.2
2,4-Dinitrotoluene	42 MS
Di-n-octylphthalate	52 MS
1,2-Diphenylhydrazine	25 / 30
Fluoranthene	11 / 17
Fluorene	29 MS
Hexachlorobenzene	29 / 35
Hexachlorocyclpentadiene	0 / 7.1
4-Nitrophenol	23 / 33
Pentachlorophenol	4.7 / 9.8
Pyrene	12 / 17
1,2,4-Trichlorophenol	29 / 35
2,4,6-Trichlorophenol	28 / 32
Run 1 Caustic Feed MS/MSD	
Benzoic Acid	4.0 MS
4,6-Dinitro-2-methylphenol	2.4 MS
2,4-Dinitrophenol	0 MS
2-Nitrophenol	12 MS
4-Nitrophenol	2.6 / 76
Pentachlorophenol	2.6776 11 MS
2,4,5-Trichlorophenol	58 MS
2,4,6-Trichlorophenol	45 MS
2,4,0-111011010prientiti	43 1013

#### 15.1.7 Blanks

<u>Requirements</u>: One method blank per batch carried through all preparation steps. Results should be less than the lowest standard.

<u>Findings</u>: Method blanks were analyzed as required. There were no positive results found in any method blank.

#### 15.1.8 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

### 15.2 SIGNIFICANT FINDINGS

There were 1535 quality control criteria evaluated for this analysis. 1453 of the criteria were found to meet the project objectives and 83 were found to be outside control limits.

All three spent activated carbon samples reported the 2,4,6-tribromophenol and the terphentl-d14 surrogates below the quality criteria. Since these were one surrogate from the acid fraction of the analysis and one surrogate from the base/neutral fraction of the analysis and all other surrogates were reported within the quality criteria, no qualification is needed. The make-up water samples G-3004-R2-Water and G-3087-Water both reported the 2-fluorophenol (3.0% & 0.87%)and phenol-d5 (0.41% & 0.0%) surrogates below the quality criteria. Both of these surrogates are from the acid fraction of the sample. There was no sufficient sample volume for re-extraction and reanalysis of these samples. All results from these samples should be considered suspect and qualified estimated biased low.

Laboratory control samples were analyzed in association with these samples with all recoveries within the quality criteria. Matrix spike / matrix spike duplicate samples were analyzed for each matrix. There were several recoveries reported outside the quality criteria indicating a matrix effect. Below is a list of the recoveries reported below the criteria. All of the results for compounds for the same matrix should be qualified as estimated biased low for recoveries below the criteria. Only the 2-methylnaphthalene and naphthalene recoveries for the spent activated carbon matrix reported high recoveries when the original result was not more than four times the spike. All three carbon samples reported positive results for these compounds and should be considered estimated biased high.

## 16.0 PROCESS METALS

The process metals data were received in a single package. Sample G-28888-R1-Carbon, G-2892-R1-Scrubber, G-2896-R1-POTW, G-2900-R1-Caustic, G-2904-R1-Water, G-2986-R2-Carbon, G-2990-R2-Scrubber, G-2990-R2-Scrubber, G-2990-R2-Scrubber, G-3077-R3-POTW, and G-3085-R3-Water were included. The samples were analyzed for total metals by SW846 Method 6010B inductively coupled plasma (ICP) and by SW846 7470 for mercury by cold vapor atomic absorption. Applicable compliance areas were reviewed and the significant findings discussed below. Section 16.1 provides a list of the primary data quality objectives evaluated during this review. Section 16.2 summarizes the significant findings of the evaluation.

### 16.1 DATA QUALITY OBJECTIVES

## 16.1.1 Contract Compliance Monitoring

<u>Requirements</u>: Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

# 16.1.2 Sample Handling Criteria

Requirements: Mercury samples are to be held no more than 28 days and ICP samples no more than 6 months.

<u>Findings:</u> All mercury samples were analyzed within 28 days and all ICP samples were analyzed within 6 months. The samples were transported to the laboratory by personnel of the stack sampling company and released to the custody of laboratory personnel. However, the stack sampling personnel did not sign the chain of custody documentation.

# 16.1.3 Initial and Continuing Calibrations

<u>Requirements:</u> GFAA and CVAA – A minimum of five standards bracketing the expected concentration. Correlation coefficient of linear plot > 0.995

ICP, GFAA and, CVAA – Continuing calibrations analyzed at the beginning and end of each analysis period and after every ten samples. Results should be  $\pm$  10% of the theoretical value for the ICP analysis and  $\pm$  20% of the theoretical value for GFAA analysis.

<u>Findings:</u> All initial and continuing calibration quality criteria were met.

## 16.1.4 Precision Objectives

Requirements: The relative percent difference between the duplicate analysis for one sample should be < 35%

<u>Findings</u>: Matrix spike /matrix spike duplicate samples were analyzed for each matrix to evaluate precision. All of the caustic feed matrix spike / matrix spike duplicate ICP RPDs exceeded the quality criteria. Sample G-2900-R1-Caustic reported positive results for barium, chromium, lead, manganese, nickel, and silver. Results for these analytes should be considered estimated.

# 16.1.5 Accuracy Objectives

Requirements: Recovery of each metal of concern spiked onto a sample should be 70 – 130%.

Calibration check standard should be 90 – 110% of the true value.

<u>Findings:</u> All calibration check standards reported recoveries within the quality criteria. Laboratory control samples were also analyzed in association with all samples with all recoveries within the quality criteria.

Matrix spike / matrix spike duplicates were analyzed for each matrix. There were recoveries for the carbon and caustic matrix spikes reported outside the quality criteria. Below is a list of MS/MSD recoveries outside the quality criteria and analytes that require qualification based on the matrix spike recoveries. All three of the spent activated carbon samples, G-2888-R1-Carbon, G-2986-R2-Carbon, and G-3069-R3-Carbon, should have the antimony and thallium results considered estimated biased low and the aluminum and barium results considered estimated biased high. All of the ICP analyte results for sample G-2900-R1-Caustic should be considered estimated biased low except, silver, which should be considered, estimated biased high.

MS/MSD Recoveries outside the Quality Criteria

### Run 1 Carbon MS/MSD

Aluminum (Al)	145 / 143
Antimony (Sb)	23 / 24
Barium (Ba)	142 MSD
Thallium (TI)	68 / 70

# Caustic Feed MS/MSD

Aluminum (Al)	70
Antimony (Sb)	64
Arsenic (As)	67
Barium (Ba)	27
Beryllium (Be)	56
Cadmium (Cd)	59
Chromium (Cr)	45
Cobalt (Co)	57
Copper (Cu)	63
Lead (Pb)	54
Manganese (Mn)	55
Nickel (Ni)	55
Selenium (Se)	63
Silver (Ag)	64 / 135
Thallium (TI)	60
Vanadium (V)	61
Zinc (Zn)	70

## 16.1.6 Blanks

<u>Requirements</u>: Analyze one method blank for each batch carried through all preparation and analysis steps, used to evaluate contamination.

<u>Findings</u>: Method blanks were analyzed as required. A positive result was reported for zinc in the method blank associated with the spent activated carbon blank. Also, several of the continuing calibration blank reported positive result. The only samples reporting positive results less than five times an associated blank were the beryllium results for the three spent activated carbon results. The beryllium results for samples G-2888-R1-Carbon, G-2986-R2-Carbon, and G-3069-R3-Carbon should be considered estimated biased high. Below is a list of the positive results reported in the blanks.

#### Blank Positive Results

Calibration Blanks 4/10/06			Calibration Blanks 4/18/06				
ICB	11:50	ΑI	10.6 B	ICB	10:59	Be	0.3 B
CCB1	12:45	ΑI	20.4 B	CCB1	1:11	Be	0.4 B
		Be	0.4 B			Pb	0.8 B
CCB2	1:40	ΑI	22.9 B	CCB2	2:24	Be	0.4 B
		Be	0.4 B			Pb	0.8 B
CCB3	2.54	Ag	1.6 B	CCB3	2:24	Be	0.4 B
		Αl	23.9 B			Pb	1.0 B
		Be	0.4 B	CCB4	3:38	Be	0.6 B
		Se	1.8 B	CCB5	4:53	ΑI	7.0 B
		TI	3.9 B			Be	0.7 B
CCB4	4:07	Ag	1.5 B	CCB6	6:07	Be	0.6 B
		Αl	25.7 B	CCB7	7:20	Ag	35.6
		Be	0.7 B			Be	0.8 B
		Pb	1.2 B			TI	4.2 B
CCB5	5:20	ΑI	32.7 B	CCB8	8:34	Ag	3.2 B
		Be	0.5 B			Be	0.6 B
		TI	4.1 B			Se	1.4 B
CCB6	6:34	Ag	5.5 B			TI	2.8 B
		ΑI	23.5 B	CCB9	9:47	Be	0.6 B
		Be	0.5 B	CCB10	10:30	Be	0.4 B
CCB7	7:35	Ag	2.0 B				
		ΑI	25.4 B				
		Be	0.4 B				

Method Blank (Carbon) Zinc 0.64 B

# 16.1.7 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

## 16.2 SIGNIFICANT FINDINGS

There were 1058 quality control criteria evaluated for this analysis. 995 of the criteria were found to meet the project objectives and 63 were found to be outside control limits.

Matrix spike /matrix spike duplicate samples were analyzed for each matrix to evaluate precision. All of the caustic feed matrix spike / matrix spike duplicate ICP RPDs exceeded the quality criteria. Sample G-2900-R1-Caustic reported positive results for barium, chromium, lead, manganese, nickel, and silver. Results for these analytes should be considered estimated.

Matrix spike / matrix spike duplicates were analyzed for each matrix. There were recoveries for the carbon and caustic matrix spikes reported outside the quality criteria. A list of MS/MSD recoveries outside the quality criteria and analytes that require qualification based on the matrix spike recoveries are located in section 16.1.5. All three of the spent activated carbon samples, G-2888-R1-Carbon, G-2986-R2-Carbon, and G-3069-R3-Carbon, should have the antimony and thallium results considered estimated biased low and the aluminum and barium results considered estimated biased high. All of the ICP analyte results for sample G-2900-R1-Caustic should be considered estimated biased low except, silver, which should be considered, estimated biased high.

Method blanks were analyzed as required. A positive result was reported for zinc in the method blank associated with the spent activated carbon blank. Also, several of the continuing calibration blanks reported positive result. The only samples reporting positive results less than five times an associated blank were the beryllium results for the three spent activated carbon results. The beryllium results for samples G-2888-R1-Carbon, G-2986-R2-Carbon, and G-3069-R3-Carbon should be considered estimated biased high. A list of the positive results reported in the blanks is located in section 16.1.6.

## 17.0 PROCESS PHYSICAL/CHEMICAL PARAMETERS

The process physical/chemical parameter data were received in two data packages. Samples G-2886-R1-Carbon, G-2984-Carbon, and G-3067-Carbon were included for the chloride analysis. Samples G-2887-R1-Carbon, G-2985-R2-Carbon, and G-3068-R3-Carbon were included for the ultimate analysis. Applicable compliance areas were reviewed and the significant findings discussed below. Section 17.1 provides a list of the primary data quality objectives evaluated during this review. Section 17.2 summarizes the significant findings of the evaluation.

## 17.1 DATA QUALITY OBJECTIVES

# 17.1.1 Contract Compliance Monitoring

<u>Requirements</u>: Sample reports are evaluated to determine that all of the required quality assurance and quality control measurements were taken.

Data packages are reviewed to determine that all required reporting has been met and that sufficient documentation has been provided.

Data packages are reviewed to determine that the analytical procedures required by the project were followed.

Findings: All sample reporting was provided as needed to confirm analytical results.

## 17.1.2 Initial and Continuing Calibrations

Requirements: Chloride— A multiple standards bracketing the expected concentration. Correlation coefficient of linear plot > 0.995

Chloride— Continuing calibrations are analyzed at the beginning and end of each analysis period and after every ten samples. Results should be  $\pm$  10% of the theoretical value.

Findings: All initial and continuing calibrations were within the quality criteria.

## 17.1.3 Precision Objectives

Requirements: The relative percent difference between the duplicate analysis for one sample should be < 10%

The PRD of the optional MS/MSD should be< 10%.

<u>Findings</u>: Each of the ultimate analyses was analyzed in duplicate and all RPDs were within the quality criteria. A duplicate and a matrix spike / matrix spike duplicate of the run 1 sample were analyzed for chloride. The MS/MSD RPD was within the quality criteria and the duplicate RPD exceeded the quality criteria. Chloride results for samples G-2886-R1-Carbon, G-2984-Carbon, and G-3067-Carbon should be considered estimated.

# 17.1.4 Accuracy Objectives

Requirements: Recovery of a known material where available should be 90 – 110%.

Recovery of optional matrix spike/matrix spike duplicate should be 90 – 110%.

<u>Findings</u>: The reference material for the chloride analysis reported recoveries within the quality criteria and the matrix spike / matrix spike duplicate reported one recovery within the criteria and one exceeding the criteria. Chloride results for samples G-2886-R1-Carbon, G-2984-Carbon, and G-3067-Carbon should be considered estimated biased high.

The instrument used to analyze the ultimate analysis is self calibrating when standards are introduced therefore no recoveries are calculated.

### 17.1.5 Blanks

Requirements: Blanks where available should be less than 20% of the sample value.

<u>Findings:</u> Blanks were analyzed as required for these methods. The method blank for chloride reported a positive result (63.0 B). All field sample results were reported more than five times this concentration. No qualification is required.

## 17.1.6 Qualitative and Quantitative Results

Requirements: The data package is evaluated to determine if there are transcription or calculation errors.

Problems with the quality of the data that could lead to inaccuracies in the data that have not been previously discussed are evaluated and discussed in this section.

<u>Findings:</u> There were no transcription errors found during the review of this data. There were no additional issues not already discussed noted.

## 17.2 SIGNIFICANT RESULTS

There were 36 quality control criteria evaluated for this analysis. 34 of the criteria were found to meet the project objectives and 2 were found to be outside control limits.

Each of the ultimate analyses was analyzed in duplicate and all RPDs were within the quality criteria. A duplicate and a matrix spike / matrix spike duplicate of the run 1 sample were analyzed for chloride. The MS/MSD RPD was within the quality criteria and the duplicate RPD exceeded the quality criteria. Chloride results for samples G-2886-R1-Carbon, G-2984-Carbon, and G-3067-Carbon should be considered estimated.

The reference material for the chloride analysis reported recoveries within the quality criteria and the matrix spike / matrix spike duplicate reported one recovery within the criteria and one exceeding the criteria. Chloride results for samples G-2886-R1-Carbon, G-2984-Carbon, and G-3067-Carbon should be considered estimated biased high.